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BULLETIN No. 61.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY.

PURE-FOOD LAWS

OF

EUROPEAN COUNTRIES

AFFECTING

AMERICAN EXPORTS.

PREPARED UNDER THE DIRECTION OF

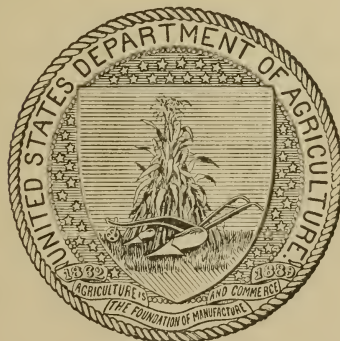
H. W. WILEY,

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WASHINGTON:

GOVERNMENT PRINTING OFFICE.

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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,

Washington, D. C., January 3, 1901.

SIR: I transmit herewith, for your inspection and approbation, the manuscript of Bulletin No. 61, of the Division of Chemistry, containing abstracts of the laws regulating the sale of food products in foreign countries.

This bulletin has been prepared, by your direction, in accordance with the provisions made by the act of Congress providing for an inspection, by the Secretary of Agriculture, of food products intended for export to foreign countries.

H. W. WILEY,
Chief Chemist.

Hon. JAMES WILSON,
Secretary of Agriculture.

INTRODUCTION.

Food products exported from the United States to foreign countries are sold in accordance with the local regulations of the several countries into which they are imported. In order that our food products may successfully meet the requirements of foreign legislation, it is important that they be inspected before shipment and a certificate of their composition be furnished for the use of the officials of foreign countries.

The Secretary of Agriculture is empowered by the Congress of the United States to conduct an inspection of this kind in an enactment which authorizes—

the Secretary of Agriculture to investigate the character of the chemical and physical tests which are applied to American food products in foreign countries, and to inspect before shipment, when desired by the shippers or owners of these food products, American food products intended for countries where chemical and physical tests are required before said food products are allowed to be sold in the countries mentioned.

In harmony with the first part of this authority, this bulletin has been prepared especially for the benefit of our exporters of foods, in order that they may know the exact conditions in which their foods must be to comply with the legal restrictions of foreign countries.

This bulletin does not assume to give the full text of all the pure-food laws of foreign countries, nor does it enter into the decisions of the courts, in the several countries mentioned, relating to the execution of these laws. It simply gives a brief summary of the points which are most important and with which our exporters of foods should be thoroughly acquainted. If the foods which are sent abroad are in condition to meet the requirements contained in this bulletin, it is not probable that they will be subjected to any hurtful restraint.

Furthermore, when the inspection of such exported foods has been thoroughly established the exporter will be furnished with an official certificate which can be presented to the officers of foreign countries charged with the enforcement of pure-food laws. Our food products on reaching foreign countries should thereby be protected from erroneous or incomplete analysis or unjust discrimination, either from the analytical or legal point of view.

The suspicion has been at times justly entertained that American food products in foreign countries have been condemned and refused sale on insufficient grounds. The inspection of our food products before shipment to foreign countries should allay this suspicion and should also result in securing greater freedom from adulteration, and this is one of the great points of advantage which should accrue from the rigid execution of the law authorizing inspection. The manufacture and sale of adulterated food products under the guise of pure foods should be prohibited whether intended for home consumption or for exportation. We can not afford to follow the example of some countries which exercise a rigid control of food products intended for home consumption, but are lenient in the control of similar food products intended for export to foreign countries. It is quite certain that we are receiving in this country many food products so adulterated as to exclude them from sale in the countries where they are manufactured. The honesty of commerce and the good character of our foods can be best conserved by requiring for our products exported to foreign countries the same freedom from adulteration, the same purity, and the same excellent condition which we expect of similar products consumed at home.

One great source of the wealth of our country is the exportation of food products. The continued prosperity of our agricultural interests depends largely on extending our foreign markets. It is evident that one of the best ways of doing this is to send to foreign countries only food products of the highest grade and above suspicion of adulteration. This bulletin, placed in the hands of our exporters of foods, will guide them in their efforts to secure this high standard of exports, and the cordial cooperation of all exporters is invited to secure to the fullest possible extent a proper execution of the provisions of the act of Congress relating to this matter.

Regulations for securing samples for inspection and for issuing certificates thereof are now in preparation and will be ready for distribution in a short time to exporters of food products (other than meat products, which are already provided for under the inspection regulations of the Bureau of Animal Industry), and to others interested in the extension of our markets for agricultural products in foreign countries. Applications for these regulations are invited. Such applications will be placed on file, and the requests will be complied with at the earliest possible moment.

H. W. WILEY,
Chief Chemist.

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PURE-FOOD LAWS OF FOREIGN COUNTRIES AFFECTING AMERICAN EXPORTS.

GENERAL SUMMARY.

With the exceptions noted below, almost any food product which is in a good state of preservation and is labeled plainly and distinctly, and in such a manner as to give a true idea of its character, may be sold in any country.

MEAT PRODUCTS.

The new German law prohibits the importation of canned meat, sausage, and macerated meat of all descriptions. Fresh meat may be imported under restrictions. The addition to meat of preservatives and coloring matter is usually prohibited.

DAIRY PRODUCTS.

The requirements of various countries regarding dairy products are very similar to those affecting meat. Butter and cheese substitutes are required to be branded according to carefully prescribed directions, and the amount of butter fat which these substitutes may contain is limited. Belgium requires that oleomargarine shall be sold uncolored, while in Holland and Denmark a maximum depth of color is prescribed.

WINE AND BEER.

Only the fermented juice of the fresh grape, subjected to the usual cellar manipulation, whose limits are carefully defined in the various countries, may be sold as wine. If any other saccharine matter or any foreign material be employed, the product must be so designated as to indicate the fact. Prohibition of the use of chemical preservatives and aniline dyes is almost universal, while the employment of all foreign coloring matter is often prohibited.

The use of chemical preservatives and foreign coloring matter with beer is usually prohibited.

CEREAL PRODUCTS.

Almost all countries require that cereal products shall be prepared from grain that is free from dirt and fungi, mineral matter, and other impurities. The mixture of the ground product of various cereals, or of cereal flour with pea flour, etc., is permitted only when properly labeled.

SUGAR, GLUCOSE, AND CONFECTIONS.

Sugar, glucose, etc., must be commercially pure and must be free from admixture with any foreign substance. Confections may be colored by harmless coloring materials (a list is usually specified), but must be prepared from pure ingredients and must be free from adulteration of any description.

ARTIFICIAL SWEETENING MATERIALS.

The sale of foods containing saccharin, sucrol, and similar preparations is prohibited in Belgium, France, Germany, Italy, and Roumania. The importation of saccharin except for medicinal use and under prescribed conditions is prohibited by Belgium and Greece.

COLORING MATTER.

All countries permit the dyeing of confections and similar articles which are themselves colorless, but are customarily colored artificially. Lists of permissible and of prohibited colors have been adopted by Austria, Belgium, France, Germany, Roumania, and Switzerland. Belgium permits mustard to be colored artificially when properly labeled. Belgium and Holland require that wine to which coloring matter has been added shall be so marked as to indicate that fact. The addition of injurious coloring matter to wine is prohibited in Denmark, France, and Tunis.

CHEMICAL PRESERVATIVES.

The sale of foods containing these substances is usually prohibited. Salicylic acid and boric acid have been used so much more commonly than others that legislation is usually directed against them, though boards of health and similar bodies which have discretion in the matter usually extend the prohibitions to benzoic acid and other preservatives as they come into use.

The sale of foods containing preservatives is prohibited in Austria, France, Hungary, and Roumania. The sale of beverages containing preservatives is prohibited in Belgium, Germany, Tunis, and Switzerland. The addition of salicylic acid to food is prohibited in Buenos Ayres and France. Holland does not permit the sale of beer containing salicylic acid, and Spain forbids its addition to wine. Italy permits the addition of 0.2 per cent of boric acid to butter, but forbids the use of other preservatives.

CONTAMINATION WITH METALS.

Strict regulations regarding the content of poisonous metals of food receptacles and utensils used in the preparation of foods have been adopted by Austria, Belgium, France, Germany, and some of the cantons of Switzerland.

AUSTRIA.

COLORING MATERIALS.

The use of colors which contain any metal except iron and the use of gamboge, picric acid, and all aniline derivatives for the purpose of coloring food and food products is forbidden.

For coloring toys, preparations containing arsenic, antimony, lead, cadmium, copper, cobalt, nickel, mercury (cinnabar excepted), zinc, or gamboge are prohibited. The use of other metallic colors for coloring toys is permitted, provided the color be coated with a waterproof varnish. The colors whose use is forbidden with toys may be employed with earthenware, provided they are covered with a glaze which is burned in.

The use of poisonous colors, such as arsenic preparations, with artificial flowers and similar substances, is forbidden unless the article be covered over with a waterproof varnish. Wall paper and similar material must not be colored with arsenic preparations.

The sale of food which has been prepared in vessels coated with poisonous colors, or stored in receptacles so coated, is prohibited. The importation and sale of wines colored with aniline dyes are prohibited. Foods and food products which are themselves white or colorless (confections, beverages, etc.), but which are ordinarily artificially colored, may be colored by any of the following substances, provided the articles so colored shall be sold from the factory only in the original packages which are distinctly labeled with the name of the material employed for coloring the contents of the package, and also with the registered seal or trade-mark of the manufacturer. The label must also bear a statement from a prescribed official laboratory (Chemischen Hochschulinstitute) that the contents of the package contain no substances deleterious to health. This statement must bear a later date than the latest decision of the health office regarding the subject and must be renewed at least annually. The list of aniline colors which may be employed under these restrictions is as follows:

Fuchsin—rosaniline hydrochlorate.

Acid fuchsin (rubin)—sodium or calcium salt of rosaniline disulphonic acid.

Rocellin—sulpho-oxyazonaphthalin.

Bordeaux red—formed by the combinations of *beta*-naphthol disulphonic acid with diazo compounds of zylol and the higher homologues of benzene.

Ponceau red—same as Bordeaux red.

Eosin—tetrabrom-fluorescein.

Erythrosin—tetraiodo-fluorescein.

Phloxin—tetrabrom-dichlor-fluorescein.

Alizarin blue— $C_{17}H_9NO_4$.

Aniline blue—triphenyl rosaniline.

Water blue—triphenyl rosaniline sulphonic acid.

Induline—the sulphonic acid compound of azo-diphenyl blue and its derivatives.

Acid yellow R—the sodium salt of amido-azo benzene sulphonic acid.

Tropæolin 000—sulpho-azo benzene- α -naphthol.

Methyl violet.

Malachite green.

Naphthol yellow.

In addition to the above, only the following colors may be added to food:

White.—Tragacanth.

Red.—Cochineal, carmine, kermes, infusion of red poppy.

Yellow.—Saffron, safflower, turmeric.

Blue.—March violet, blue bottle, indigo, prussian blue, ultramarine, sea blue (form of artificial ultramarine).

Green.—Spinach juice.

Violet.—Cochineal infusion with lime water.

Gold.—Pure gold leaf.

Silver.—Pure silver leaf.

Wrappers for confections, coffees, and other varieties of food must either be white or prepared from material which is naturally colored. If a wrapper which is artificially colored be employed, a second wrapper of the character above described must be placed between it and the inclosed product, and no artificially colored wrapper may be used in any case to inclose any but a dry, solid material. The use of wrappers containing copper salts is especially prohibited.

RECEPTACLES.

Food receptacles and utensils intended for the preparation of food must not be either partially or entirely composed of an alloy containing more than 10 parts of lead per 100 parts of the alloy. The inside of such receptacles must not be coated with tin which contains lead. Such receptacles must not be soldered with an alloy containing more than 10 per cent of lead. In case of glazed and enameled ware, lead must not be present in such state that it will be dissolved by boiling one-half hour with a 4 per cent solution of acetic acid. The glass or enamel must not be so attached to the vessel that it will scale off. Metallic parts of nursing bottles must not contain more than 1 per cent of lead. Metal foil, which is used as a wrapper for such products as snuff and tobacco, must not contain more than 1 per cent of lead. Vessels which have been cleaned with the aid of leaden shot must not be used as receptacles for food products. The sale of food products which have been ground with millstones filled with lead or an alloy containing lead is prohibited.

Rubber or caoutchouc which contains lead or zinc must not enter into the composition of such articles as nipples of nursery bottles, rubber rings, nipple shields, etc., or as receptacles for such articles as beer, wine, vinegar, and preserves, or of vessels which are to be used in the preparation of food products or as receptacles for the same.

If antimony sulphid enters into the composition of vessels which are used in connection with food products, it must be so prepared that no antimony is dissolved by a dilute solution of tartaric acid. Copper and brass vessels must not be used in the preparation of foods unless

the inner side be coated with lead-free tin. All manipulations are prohibited which could by any means bring copper compounds into the composition of food materials.

The addition of fluorids to foods is especially prohibited, as is also the addition of salicylic acid to wine.

MUNICIPAL REGULATIONS OF VIENNA.

Municipal regulations for Vienna prescribe that the term "butter" shall be used only for the exclusive product of pure milk or cream. Fats from all other sources must be designated as margarine butter, lard, or compound lard, according to their character. Margarine butter must be molded in brick-form prints, and the words "Margarinebutter" must be marked on every print in distinct characters of such size that the words shall extend the entire length of the print. The wrapper in which each print is sold must also be marked in distinct indelible characters with the words "Margarinebutter." Every receptacle containing compound lard must be distinctly printed with the name "Margarineschmalz" or "Kunstfett." The terms "Echtebutter" or "Butterschmalz" are applied only to articles containing fat obtained from pure milk. "Schweinefett" must be used only to designate pure lard. "Margarinebutter" is applied to all butter substitutes which do not consist exclusively of butter fat. "Kunstfett" is used to designate compound lard.

BELGIUM.

EDIBLE FATS.

The word "lard" must be applied only to pure unmixed swine fat. All other edible fats, excepting butter and margarine, must be so marked as to indicate exactly their origin, or with the words, "mixed fat" (*graisse mélangée*).

All receptacles containing other edible fats than lard, butter, and oleomargarine, must be plainly marked as described above, and also with the name of the manufacturer or dealer, or with some registered mark.

Lard and other edible fats which contain more than 1 per cent of water or salt must be labeled, "watered" (*aqueux*), or "salted" (*salé*). The addition of mineral substances, other than salt, and of chemical preservatives and glycerin is forbidden.

It is forbidden to sell spoiled or deteriorated edible oils as food. All receptacles containing oils must be branded with the word "oil" immediately preceded by a word in similar type which will give the true and exact source of the contents of the receptacle; for instance, olive oil, peanut oil, sesame oil, etc.

BUTTER.

The term "butter" must be used only with reference to fat obtained exclusively from milk or cream with or without the addition of coloring matter or salt. All butter containing other additions and all butter substitutes must be designated as margarine. Margarine must not contain more than 5 per cent of butter fat and must not be artificially colored. The maximum color permitted in margarine may be decided by the minister of agriculture. These regulations regarding the addition of butter fat to margarine and the height of color of the same are not applied to margarine intended for export from Belgium.

The receptacles and packages which contain margarine must be plainly labeled with the word "margarine" in letters at least 2 cm high, as well as the name of the manufacturer or dealer. Margarine which is not in packages must be molded in cubical form with the word "margarine" impressed, as well as the name of the manufacturers or dealers. The sale of rancid butter or butter made from the milk of diseased or improperly fed cows is forbidden. It is also required that margarine shall be fresh and made from the fat of healthy animals. The addition of glycerin to butter and margarine is prohibited.

COCOA AND CHOCOLATE.

The term "cocoa mass" must be used exclusively for the product of the seed of the cocoa tree, whether it be raw or roasted, entire, hulled, or ground, with, or without the addition of foreign substances. Finally, such product may be melted or molded in ingots or tablet form or pulverized. The term cocoa may be applied to the prepared product of the cocoa tree from which a portion of the fat has been removed, provided that the fat content of the product is not less than 20 per cent. The term "alkalized cocoa" may be used to describe the product to which an addition of alkaline carbonate has been made to render it more soluble; but the alkaline carbonate so added must not exceed 3 per cent of the total weight of the product. Cocoa which contains more than 3 per cent of alkaline carbonate is considered unwholesome and its sale is forbidden. The characterization "alkalized" is not necessary if the product is intended for export from Belgium.

Cocoa which is prepared otherwise than by the methods described above must be marked on the wrapper with the word "cocoa," followed in the same type by words which will give an exact description of the method used in preparation. The term chocolate is applied to the product made exclusively from hulled cocoa, to which at least 35 per cent of its weight of cane sugar has been added, with or without the addition of spices.

Products which contain 35 per cent of hulled cocoa, but at the same time other substances than sugar and spices, can be sold only when

marked on the wrapper in the same type as the word "chocolate" with a word which will give an exact description of the foreign substances present, or when labeled with a name in which the word "chocolate" does not appear. When molded in tablet form, the above description must be impressed or printed in raised characters on every tablet. Any preparation which contains less than 35 per cent of hulled cocoa must not be sold as cocoa bon bons or under any other name in which the word "cocoa" or "chocolate" appears. All bills and shipping receipts must be designated in the same manner as the preparations described above. All packages of cocoa must be marked with the name of the manufacturer or dealer or with the registered mark. These provisions apply to ordinary chocolate in tablet, block, or powdered form, or chocolate croquettes, but not to special preparations containing chocolate sold by confectioners and bakers.

CHICORY.

The term "chicory" must be applied exclusively to the product of the chicory root, either in its natural condition or by any appropriate treatment, such as roasting, powdering, drying, etc. Chicory must not contain more than 15 per cent of water (dried at 100° C.). The ash content of the dried material must not exceed 10 per cent when finely powdered, or 8 per cent when coarsely powdered. Chicory must not lose more than half its weight when extracted with boiling water. Chicory which is put up in packages, with the weight of the contents marked on the package, may have a higher water content than 15 per cent if the weight of substance in the package is correspondingly greater than that stated on the label. An addition of fat or saccharine matter not exceeding 2 per cent of the total substance is permitted. Bags and other receptacles in which chicory is shipped or sold must bear the name of the packer or dealer, or some registered mark.

MUSTARD.

The sale of any substance other than a mixture prepared of ground black and white mustard seed, under the unqualified name of "mustard," is prohibited. All similar preparations, such as those containing pepper, estragon, rice, and foreign coloring matter, can be sold only when each package bears in the same type as the word "mustard" the names of all foreign substances present, or the designation "prepared mustard," or some designation not containing the word mustard may be employed. In the preparation of mustard the use of vinegar which does not comply with the law of January 3, 1894, is prohibited. The use of deteriorated, decayed, or unwholesome substances in manufacturing prepared mustard is forbidden. Mustard preparations which do not comply with these requirements and are not intended for use

as a condiment must be plainly labeled with a statement of the use for which they are intended. All packages of mustard and mustard preparations must be marked with the names of the manufacturers or dealers or with a registered label.

FISH.

Fresh or preserved fish which has been mixed with matters other than spices, condiments, aromatic jellies the principal ingredient of which is gelatin or gelose, must not be sold unless a plain label shall indicate the nature of the foreign substance used. Canned-fish products must have a label showing the kind of fish, and also, if necessary, the kind of oil, etc., used. Fish, shellfish, etc., caught with indian berry (*Cocculus indicus*) or other poisonous substances and those mixed with antiseptics are declared injurious. No substances injurious to health are allowed to be used. Receptacles containing fish must bear the name and address or the registered mark of the seller. It is further forbidden to sell or keep in the same premises with food products fish not intended for alimentary purposes unless these are clearly marked "Not eatable," or the like.

SUGAR.

It is provided that the word "sugar" and similar terms shall refer only to the product obtained from the juice of sugar cane, sugar beet, and similar plants. All other products, such as dextrose, which are used for sweetening purposes must be properly labeled. Mixtures of cane sugar with other materials, such as dextrose, can be sold only when so labeled as to inform the purchaser of the character of the goods.

White sugar must not contain more than 0.2 per cent of mineral substances, raw sugar not more than 2.5 per cent of mineral substances, and glucose not more than 0.8 per cent of mineral substances. Glucose must not contain more than 0.05 grams of free acids (calculated to sulphuric acid) per 100 grams of dry matter, nor appreciable quantities of oxalates, oxalic acid, arsenic compounds, lead, zinc, or barium.

Sugar must not be deteriorated in any manner—for instance, coated with mold. The addition of preservatives and the presence of fungicides are forbidden. Bags, barrels, and other receptacles must be plainly marked with the name of the manufacturer or dealer.

SACCHARIN.

The importation, manufacture, shipping, and selling of saccharin and other products, which are formed synthetically and possess a sweet taste similar to that of sugar but have no nutritive value, are prohibited. The use of saccharin and similar products in the preparation of foods and the sale of foods containing them are also prohibited.

FLOUR AND BREAD.

The words "flour" and "bread" must be used exclusively to denote wheat products. For designating the product of any other cereal it is necessary to employ also the name of that cereal, for instance, "rye flour," "rye bread," etc. Mixtures of rye flour with other cereals must be designated by the word "môteil." Flour must be manufactured from grain which is sound and in good condition and which has been thoroughly cleaned. The sale of flour which is adulterated with mineral matter is prohibited. The word "tapioca" must be used exclusively to refer to food products derived from the cassava root.

WINE.

In the application of these regulations one understands—

(1) By wine, the product of alcoholic fermentation of the juice or must of the fresh grape.

(2) By sweet wines or liqueurs or cordials ("vin de liqueur" or "vin de dessert"), the product of alcoholic fermentation, whether it be of the juice or must of the grape, more or less dried, or concentrated by evaporation, containing usually about 14 to 18 per cent of alcohol and an excess of natural grape sugar.

(3) By sparkling wines (vin mousseux), the product of the fermentation of the juice or must of the fresh raisin surcharged with pure carbonic acid.

(4) By wine of the second vat, wine made from the residuum of grapes (piquette), wine from the lees or dregs, wine from the dried grape, sparkling wine from the dried grape, cider, sparkling cider, hydromel, etc., the vinous beverages which present an analogy with wines and which are the product of the fermentation of the juice or must extract of the dregs or lees of the fresh or dried grape, of the juice of the apple, of honey, etc., with or without the addition of sugar, alcohol, or pure carbonic acid.

It is forbidden to sell or expose for sale, to hold, or transport for sale or for delivery as wine, any wine to which foreign substances have been added.

This prohibition does not apply to the following:

(1) The addition of clarifying agents acting mechanically (albumin, gelatin).

(2) The addition of ordinary salt on condition that the content of chlorids, calculated as sodium chlorid, does not exceed 2 grams per liter.

(3) The addition of gypsum on the condition that the content of sulphates, calculated as potassium sulphate, does not exceed 2 grams per liter.

(4) The presence of sulphurous acid, because of sulphuring the casks, on condition that the wine shall not contain more than 2 milligrams of free sulphurous acid nor more than 20 milligrams of total sulphurous acid per 100 cc.

(5) The addition of pure sugar or alcohol, provided that the receptacles in which the wine is placed shall bear in a conspicuous place and in plain characters, as large and as conspicuous as any other letters used for other inscriptions, the word "sugared" or "alcoholized" ("sucré" or "alcoolisé"), as the case may be, and that this statement be reproduced on the invoice, the bill of lading, or the booking-office ticket.

Wine, as well as the vinous beverages having an analogy to wine, to which have been added foreign substances, with the exception of those enumerated above, can not be kept for sale, exposed for sale,

for delivery or retail, except in receptacles bearing in a prominent place and in legible characters, as large and as conspicuous as those employed for any other inscription, an indication of the materials introduced in their preparation; for example, "watered wine," "colored wine," "aromatized wine," "dried grape wine," "cherry wine," or an inscription sufficiently clear to make known their origin, such as "piquette," "eider," "hydromel." This statement need not include the names of the vineyards of true and natural wines. These should be found in the invoices and the bills of lading or booking-office tickets.

Wines, liqueurs (vins de liqueurs), sparkling wines, and vinous beverages to which the following substances have been added are declared injurious:

Ethers, or essential oils (oil of wine);

Bitter almond, cherry, laurel;

Alkaloids;

Compounds of arsenic, lead, zinc, aluminum, barium, strontium, calcium, magnesium, alkalies;

Mineral acids, free or combined oxalic acid;

Salicylic acid or other antiseptics (with the exception made in favor of sulphurous acid in the amount specified);

Glycerin;

Sugars, cask sugars, or impure alcohol, the sale of which is forbidden for edible purposes by the rules relative to those commodities; alcohols other than ethyl alcohol;

Sulphates, in greater quantity than indicated above, or of more than twice that quantity in the case of liqueurs (vins de liqueurs).

It is forbidden to add to wine or liqueurs (vin de liqueur), to sparkling wines, or vinous beverages, any of the substances mentioned above, or any other substance injurious or dangerous to the health.

All casks in which wine, liqueurs, and vinous beverages will be exposed for sale or delivered must bear the name of the firm, as well as the address, or at least the registered mark of the maker or seller.

DENMARK.

WINE.

The following additions to wine are prohibited:

Alum, or other soluble aluminum salts; barium compounds; strontium compounds; magnesium compounds; boric acid; salicylic acid; spirits containing fusel oil; crude (not technically pure) glucose; kermes; injurious coloring material; glycerin; saccharin; flavoring materials, such as ethereal oils, essences, etc.; gums, and other organic and inorganic materials intended to increase the extract content.

The following additions are permitted without declaration:

The use of common clarifying agents, such as albumin, gelatin, isinglass, Spanish earth, and other common substances; the neutralization of excessive acid with precipitated calcium carbonate; the customary sulphuring of casks; the pasteurization of wine; the blending of wines (in blending only dry wines may be mixed with dry wines).

Dry wines must not contain more than 0.2 gram of sulphuric acid (calculated to potassium sulphate) per 100 cc. The addition of foreign coloring matter is prohibited unless the same is declared on the label. The addition to dry wines of saccharine matter either in a solid state or in solution is permitted if the same is stated on the label. The same is true of the addition of water. These provisions do not apply to red wines which are rich in extract and coloring matter and hence in their natural state not suitable for consumption, provided that after treatment such wines shall not contain less than 2 grams of sugar-free extract per 100 cc, and that no sugar other than the ordinary grape sugar shall be found in the extract. Wines which shall receive an addition of water and which fulfill the required conditions of percentage of extract, etc., may be blended with other wines of normal composition without regard to the extract content of the blend so produced. The addition of alcohol to dry wine must be indicated on the label; this, however, does not apply to the alcohol necessary for ordinary cellar manipulation. The alcohol so employed must be fully refined and of not less than 93.25 per cent by volume, and the amount added must not exceed 2.5 liters for 240 liters of wine. In the case of wines which are not fully fermented and whose sugar content is such as to make it doubtful whether they should be classified as dry or as sweet wines, the addition of alcohol of not less than 93.25 per cent per volume in such quantity that the alcohol content of the product shall not exceed 17 per cent per volume is permitted. Port wine, sherry, madeira, and liqueurs from foreign lands must conform to the customary composition of these wines in the country where they are produced. These wines may be manufactured from dried grapes under the condition that the alcohol content shall not exceed 25 per cent per volume, and the sugar-free extract shall not be less than 2 grams per 100 cc. On the other hand, the addition to these wines of sugar or other material which is not the product of the grapes, without indicating the same on the label, is prohibited. Wines of this class which are too low in alcohol may be fortified with alcohol of not less than 93.25 per cent by volume. The alcohol content of the product must not exceed 25 per cent by volume. Dessert wines must be the customary product of the region of their production with the exception that they may receive the ordinary cellar manipulation. The term "champagne" may be applied only to wines fermented under pressure. Carbonated wines may be sold if properly designated.

Cognacs, rum, and arak must not receive the addition of alum or other soluble aluminum salts, barium compounds, strontium compounds, magnesium compounds, boric acid, salicylic acid, alcohol containing fusel oil, crude glucose, kermes, or other unwholesome materials.

OLEOMARGARINE.

This product must be branded and put up in prints in a prescribed manner; it must not contain more than 50 per cent butter fat, and the shade of color permissible is fixed.

ENGLAND.

All adulterated or impoverished articles of food must be in packages conspicuously marked with the true description of the contents of the package. The addition to foods of coloring materials and preservatives which are harmless in the quantity employed is permitted.

It is required that margarine, filled cheese, etc., be conspicuously marked on the top and sides of each package with the words "margarine" or "margarine cheese," as the case may require. Margarine must not contain more than 10 per cent of butter fat. Adulterated or impoverished butter, other than margarine, must be in packages so marked as to indicate the exact nature of the contents of the package.

Every can of condensed, skimmed milk must have a label clearly visible to the purchaser, on which the words "machine-skimmed milk" or "skimmed milk," as the case may require, are printed in large, legible type.

FRANCE.

The law of February 2, 1899, regulates the commerce in fertilizers, butter, and wines especially; it also applies to all articles of merchandise of whatever nature. Misrepresentation concerning the nature, quality, or quantity of articles covered by this law is prohibited.

Cans and similar receptacles containing food must not be coated with an alloy containing more than 0.5 per cent of lead or 0.01 per cent of arsenic, and must not be soldered with an alloy containing more than 10 per cent of lead or 0.01 per cent of arsenic.

Only lead-free tin foil may be used as wrappers for food materials.

BUTTER AND BUTTER SUBSTITUTES.

The term butter shall be applied only to products made exclusively from milk or cream. All other fat materials having the appearance of butter must be sold as margarine, and must not contain more than 10 per cent of butter fat. The receptacle containing oleomargarine must be indelibly branded with the word "margarine" or "oleomargarine." The constituents of the contents of the receptacle and the percentage of each constituent present must be given on all bills rendered for such goods. In wholesale trade, the name and address of the manufacturer must be given on the receptacle containing margarine. If sold at retail, margarine must be in cubical prints with the word "margarine"

or "oleomargarine" impressed on one side of the print. Each print must also be inclosed in a wrapper on which the word "margarine" or "oleomargarine" is indelibly printed. Every bill, letter, and package in any way relating to the sale or transportation of margarine must be distinctly marked with the word "margarine" or "oleomargarine."

WINE.

The addition of sulphuric acid, nitric acid, hydrochloric acid, salicylic acid, boric acid, and analogous substances, as well as the addition of coloring matter, is prohibited. Wine must not contain more than 0.1 gram of sodium chlorid per 100 cc, or more than 0.2 grams of potassium sulphate.

Wine is defined as the fermented juice of the grape treated in no way except by the ordinary cellar manipulation, including the addition of sufficient water to the must to reduce its sugar content to 29 grams per 100 cc, or the dilution of sufficient pure alcohol to give a normal composition to very low wine. The addition of both alcohol and water to the same must or wine is not permitted under any circumstances.

The product of the fermentation of the lees, with or without the addition of sugar, and mixtures of the same with wine, can be sold only as "Vin de marc" or "Vin de sucre," and receptacles in which the same is sold must be conspicuously labeled with an orange-colored label containing the appropriate name.

The product of the fermentation of dried raisins, and mixtures of the same with wine, can be sold only as "Vin de raisins sec," and must bear in a conspicuous place a label of green paper marked with its correct name.

COLORING MATERIALS.

Foods and food products must not be colored with any mineral substance, except that prussian blue, ultramarine, chalk, and ochre may be used with confections or similar products. Confections and other products must not be inclosed in wrappers which are colored with the prohibited substances. All confections inclosed in packages must bear the name and address of the manufacturer or dealer. The use of litharge, lead acetate, and similar compounds for clarifying saccharine products and fermented beverages is forbidden.

The use of the following coloring materials with foods is prohibited:

Mineral colors:

Compounds of copper, lead, arsenic, and mercury, and barium chromate.

Organic colors:

Gamboge; aniline derivatives, such as fuchsin, Lyon blue, flavanilin, methylene blue; phtaleins and their derivatives, such as eosine, erythrosin; nitro compounds, such as naphthol yellow and Victoria yellow; diazo compounds, such as tropeolins and xyloidine red.

As exceptions to the above general regulations, however, the following compounds may be employed in coloring confections, pastry, and liqueurs, which are ordinarily white or colorless:

Rose colors:

Eosine (tetra brom-fluorescen).

Erythrosin (methyl and ethyl derivatives of eosine).

Bengal rose, phloxin (iodin and bromin derivatives of fluorescen).

Bordeaux red and Ponceau red (resulting from the action of the sulpho-derivatives of naphthol on the diaz xylens).

Acid fuchsin (without arsenic and prepared by the Coupier method).

Yellow colors:

Acid yellow (derivatives of sulphonates of naphthol).

Blue colors:

Lyon blue, light blue, Coupier blue, etc., (derivatives of triphenil rosanilin or of diphenylamin).

Green colors:

Mixtures of blue and yellow named above.

Malachite green.

Violet colors:

Paris violet or methylanilin violet.

GERMANY.

MEAT.

A new law regulating the preparation, importation, and sale of meat and meat products was passed by the Bundesrath and the Reichstag in June 19, 1900, to take effect in April, 1901. Regulations for its enforcement have not yet been promulgated. The importation, except in "free ports," of meat in hermetically sealed cans and similar receptacles, and of sausage and macerated meat of all descriptions, is unequivocally prohibited.

It is provided that fresh meat must be imported in the entire body or in halves. The meat must be so dressed that the breast, diaphragm, lungs, heart, and kidneys, and, in the case of cows, also the udder, retain their natural position in connection with the body.

Prepared and preserved meat can be imported only when the method of preparation or preservation to which it has been subjected is such as to add to or produce in the meat no injurious substances.

The above requirements do not apply to corned beef, ham, bacon, or casings provided that the corned beef is not imported in pieces weighing less than 4 kilograms (8.8 pounds). Meat which has been preserved by processes which will enable it to retain all of the characteristics of fresh meat (refrigeration) is subjected to the restrictions applied to fresh meat.

The foregoing regulations are to remain in force until December 31, 1903, or until other regulations are provided.

Horse flesh can be imported only when so designated in the German language that its true nature will be understood by the purchaser.

In Prussia a regulation is in force relating to the amount of flour that may be added to the several varieties of sausage. "Fleischwurst" shall receive at the most 4 per cent. "Blutwurst" and "Leberwurst" selling for not more than 0.70 marks per half kilogram shall not contain more than 5 per cent of flour. "Plockwurst," "Cervelatwurst," "Salamiwurst," "Bratwurst," "Mettwurst," "Blutwurst" and "Leberwurst" which sell for more than 0.70 marks per half kilogram must not receive the addition of flour. Sausages which are treated with flour must be so marked as to indicate that fact ("Wurst mit Mehlzusatz").

BUTTER AND EDIBLE FATS.

All packages of butter substitutes, filled cheese, and compound lards must be branded "Margarine," "Margarinekäse," and "Kunstspeisefett," respectively. Each package must also be marked in a conspicuous place with a red stripe at least 2 cm wide for packages 35 cm high or less and 5 cm wide for higher packages. The same articles, when sold at retail, must be in wrappers marked "Margarine," etc., and also with the name of the dealer. All prints must be cubical in form and stamped "Margarine," etc., in sunken letters.

To facilitate the examination of samples, the Bundesrath has decided that all fats used in the preparation of butterine shall receive an addition of 10 per cent of their weight of sesame oil, and all fats used in the preparation of filled cheese shall receive an addition of 5 per cent of their weight of sesame oil. The sesame oil employed must be such that when a mixture of 0.5 part of sesame oil with 99.5 parts peanut or cotton-seed oil be shaken with an equal volume of hydrochloric acid (specific gravity 1.19) and a few drops of a 2 per cent alcoholic solution of furfurol a marked red color is imparted to the acid layer.

Patterns of labels to be employed with butter substitutes, etc., have been adopted by the Bundesrath thus: The space within the line inclosing the label must not be more than 7 times as long as high, and must not be less than 30 nor more than 50 cm high, except that with round or oval packages whose greatest diameter does not exceed 15 cm the space may be decreased to 15 cm. Directly above this label a red strip at least 2 cm wide on packages up to 35 cm high, and at least 5 cm wide on higher ones, must extend around the package, but shall not interfere with the mark "Margarine," etc. The name of the manufacturer and the brand must be near the word "Margarine," but must not be in contact with it nor with the encircling line or red band. The designation, name of manufacturer, and brand must either be burned in or painted on white or bright yellow ground in black letters, and must be on two opposite sides of package and also on the top, if there be a top, and on both ends of casks. In prints, the pattern described above must be followed, but the limitation of size is removed, and the word

“Margarine” may be divided in two and the word “Margarinekäse” in three portions connected by hyphens.

In Prussia the terms “Smalz,” “Bratensmalz,” “raffinirtes Smalz,” etc., can be applied only to pure lard. Mixtures containing other fats or oils must be called by such name as “Speisefett.”

COFFEE.

Coffee substitutes must be inclosed in packages which bear a label stating the chief ingredients in combination with the word “Kaffee.” The name of the manufacturer must also be stated on the package. Mixtures of coffee and coffee substitutes can be sold only in packages which are plainly marked so as to give the purchaser a true idea of the nature of the contents, for instance, “Coffee and coffee-substitute mixture” (Kaffee-surrogat-mischung). The name and location of the manufacturer must also be stated on the package, as well as the materials from which the product is prepared.

It is forbidden to manufacture, sell, or hold for sale machines for the preparation of artificial coffee beans.

SACCHARIN.

The manufacture and sale of foods and beverages containing artificial sweetening material (saccharin, dulcin, etc.), are prohibited.

WINE.

The law prohibits the addition to wine, wine-like, or wine-containing beverages of soluble aluminum salts, barium compounds, boric acid, glycerin, kermes, magnesium compounds, salicylic acid, impure alcohol, glucose (not commercially pure), strontium compounds, and aniline dyes; or the addition of more than 0.2 gram per 100 cc. of potassium sulphate, except in dessert wines (southern sweet wines) of foreign origin. The use of “sugar water” and “pressed” grapes; of sugar and wine yeast; of raisins, currants, and other sweetening materials than cane sugar or dextrose; of acids and flavors; of gums and other substances which influence the extract, except as hereafter provided, is prohibited unless the goods are so labeled as to indicate such additions. Raisins may be added to dessert wines (southern sweet wines). The addition of saccharin is forbidden for all wines and similar beverages. More liberty is given in sparkling wines.

The following additions are permitted:

Alcohol, not over 1 per cent by volume; small amount of clarifying agents (albumen, gelatin, isinglass, etc., sodium chlorid, carbon dioxid, and sulphur dioxid); the blending of wines; neutralization with pure precipitated calcium carbonate; addition of such amounts of technically pure sucrose, invert sugar, and dextrose as will not bring the

ratio of ash to extract below that of unsugared wines of the vicinity. The extract content must not be below 1.5 grams per 100 cc; the extract content less total acids must not be below 1 gram per 100 cc; the extract content less fixed acids must not be below 1.1 grams per 100 cc. The ash must not be below 0.14 gram per 100 cc.

UTENSILS, TOYS, ETC.

Cooking utensils and receptacles for foods and vessels used for preparation of beverages and fruit juices must not contain over 10 per cent of lead in any part. The inside must not be coated with an alloy which contains over 1 per cent lead, and solder exposed to contents must not contain over 10 per cent of lead (except solder with lead-free Britannia metal). Enamels and glazes must not yield lead on boiling one-half hour with a 4 per cent solution of acetic acid. Alloys containing over 1 per cent of lead must not be used in siphons for carbonated beverages or for metal parts of nursing bottles. Rubber containing lead or zinc must not be used for mouthpieces, nursing bottles, nipple shields, etc. Rubber containing lead must not be used for drinking cups or toys (except large balls), or for tubes for beer, wine, or vinegar. Containers must not be cleaned with shot. Snuff, chewing tobacco, and cheese must not be wrapped in foil containing over 1 per cent lead. Cans must not contain over 1 per cent lead on the inside or have exposed solder containing over 10 per cent of lead.

COLORING MATERIALS.

The following are provisions relating to the addition of coloring matter to foods, beverages, toys, cosmetics, and vessels, wrappers, and covers for foods:

The addition of the following to articles of food and drink are prohibited: Colors which contain antimony, arsenic, barium, lead, cadmium, chromium, copper, mercury, uranium, zinc, tin, gamboge, corallin, and picric acid.

Vessels, wrappers, or covers dyed with the above-mentioned colors must not be used for holding or protecting articles of food or drink. This regulation does not apply to the use of the following: Barium sulphate (heavy spar, permanent white), barium colors free from barium carbonate, chrome green, copper, zinc, tin, and their alloys, when applied as metallic colors, cinnabar, tin oxid, tin sulphid in the form of gold-bronze ("musivgold") all vitrified colors in glass, glazes or enamels, and colors on the outside of water-tight vessels.

In the manufacture of toys (including picture cards, picture books, and water colors, flowerpot covers, and artificial Christmas trees) the materials mentioned above as forbidden are not to be used. This regulation does not apply to the articles enumerated above as exceptions, nor to antimony sulphid and cadmium sulphid applied as color in

gum; lead oxid in varnish; white lead as a component of the so-called molded wax, if the same does not amount to more than 1 part in 100; lead chromate by itself or in association with lead sulphate, in oil or lacquer, covered by lacquer or varnish; zinc colors insoluble in water, in rubber toys, if used in the coloring of the rubber, or as lacquer or oil color applied with lacquer or varnish, and all vitrified colors applied with enamel.

HUNGARY.

ALCOHOLIC BEVERAGES.

The addition to alcoholic beverages of strong commercial essences with a sharp odor, especially of sharp spices and vegetable materials, such as pepper, paprika, sea onions, etc., of narcotic substances, fusel oil, or any other substance that will increase its sharp or narcotic taste, is prohibited. This prohibition does not extend to medicinal and dietetic alcoholic preparations.

The manufacture and sale of adulterated wine is prohibited. All wines are considered adulterated which are not exclusively made from grape must, with such additions as are necessary in ordinary cellar manipulation. It is also prohibited to misrepresent the location in which a wine was made or the variety of grapes used in its manufacture.

The must may receive additions of refined sugar, grape sugar, or fruit sugar, as well as dried berries and dried raisins. In the Toquay wine region these additions are not permitted, but since it is fraudulent to designate wines falsely as to the place of manufacture or the variety of grape used, this prohibition does not affect wines from foreign countries.

The addition of refined alcohol and pure cognac is also permitted, and the must may be treated with arsenic-free sulphur and the scum removed by skimming. The excessive acidity may be neutralized with calcium carbonate or potassium carbonate. In no case, however, is any addition permitted which will change the composition to an appreciable extent or cause its ingredients to vary from the required proportions. The wines in cellar manipulation may receive an addition of refined alcohol or cognac, or the usual harmless clarifying agents. The acidity may be regulated, in the case of excessive acidity, by the addition of calcium carbonate or potassium carbonate, or the acid may be increased by the addition of cream of tartar, tartaric acid, or malic acid.

Wine may also be sulphured with arsenic-free sulphur and receive the proper manipulation for its preservation, providing that no injurious substance be added. In the manufacture of sweet wines refined sugar, saccharine material, caramel, dried raisins, and the required amount of yeast may be added for the after fermentation. In no case may anything be added in such quantities that the required propor-

tions of the various ingredients of the wine shall be altered. The addition to must or wine of material not specified, or especially of saccharin, glycerin, salicylic acid, flavoring extracts, ethereal oils, or other liquids, and of all vegetable, mineral, and aniline colors, with the exception of safflower, is expressly prohibited.

Carbonated wines can be sold only under the proper designation. The lees may be used in the manufacture of "Tresterwein" when they are extracted with sugar water for fermentation, and for "Nachwein" when extracted with water and refined alcohol or cognac.

ITALY.

DAIRY PRODUCTS.

The term "butter" must be used only to designate fatty material obtained from milk and cream by mechanical operations. The sale of abnormal or rancid butter or butter manufactured from the milk of diseased or improperly fed cows is prohibited. Butter must contain no injurious coloring matter, and must contain no added substances, such as foreign fats, flour, sirups, chalk, plaster, or soluble glass. No chemical preservatives may be added other than common salt and borax, and the latter must not be present in greater quantity than 0.2 per cent. The fat content of butter must not be less than 82 per cent. All edible fats which are to be used as butter substitutes, and all butter adulterated with foreign fat, must be sold under some such name as "margarine." Butter and other edible fats of animal or vegetable origin must be in a good state of preservation, and if of animal origin must have been prepared from a healthy animal.

Cheese must contain no substance which is not obtained from milk and cream, other than salt and harmless coloring matter.

The sale of eggs which are tainted or colored with injurious substances is forbidden.

CEREAL PRODUCTS.

Cereals and mill products must be in a good state of preservation, free from mold, weed seed, and other impurities. The addition of alum, copper sulphate, zinc sulphate, talc, chalk, plaster, and other impurities of all descriptions is forbidden.

SUGAR AND CONFECTIONS.

The word "sugar" is employed to designate the product of the sugar cane or sugar beet. It must not contain more than 5 per cent of reducing sugar. Sirups, confections, marmalades, etc., must not be fermented nor in any way deteriorated, and must not contain any other fruit product than that which is represented to be present, nor any toxic material, such as saccharin, glycerin, oxalic acid, nor such preservatives as boric acid and salicylic acid.

BEER.

Beer must be made exclusively from the malt of barley or other cereals, with the addition of hops, yeast, and water. The sale of beer which has become spoiled or deteriorated from any cause is prohibited. The sale of liqueurs and distilled liquors containing hydrocyanic acid, mineral acids, toxic metals, injurious colors, methyl alcohol, picric acid, gamboge, or medicinal drugs is prohibited.

VINEGAR.

The term "vinegar" is applied exclusively to the fermented product of wine. It must not contain less than 4 per cent of acetic acid, and there must be no addition of coloring matter or other substances. Vinegar obtained by the acetic fermentation of beer, cider, or alcohol may be sold if properly designated "beer vinegar," etc. The sale of vinegar which has become spoiled or deteriorated on account of age is prohibited. No free acids, such as sulphuric, hydrochloric, nitric, oxalic, and tartaric, and no bisulphite must be present.

COFFEE, TEA, AND CHOCOLATE.

The sale of coffee substitutes and adulterated coffee, or coffee colored by injurious substances, is prohibited. Tea must contain no addition of any foreign substance whatever. Chocolate must receive no addition of chalk, ocher, or other mineral matter, or indigestible or injurious vegetable substances.

MEAT AND FISH.

The Italian law requires that prepared meats shall be inclosed in a wrapper on which the kind of animal from which the meat was prepared is plainly stated. It is also required that all meats, blood, etc., used in the preparation of sausage and other meat products must be in a good state of preservation. The mixture with lard of fat from any other source than swine is prohibited.

The addition of coloring matter to fish, mollusks, and crustacea in order to give stale articles a fresh appearance is prohibited.

MUNICIPAL REGULATIONS OF MILAN.

The municipal regulations of Milan prohibit the addition of coloring matter of any kind to foods and beverages which normally are colored. In confections and other foods artificially colored, the coloring matters condemned by the German law are prohibited, and all others except certain specified colors. The addition of salicylic acid to beer is also prohibited.

ROUMANIA.

GENERAL PROVISIONS.

It is forbidden to adulterate food by the addition of foreign materials, by removing characteristic ingredients, or by any change of composition or character whereby the product is made less nutritious, less wholesome, or cheaper. The sale of unripe or decayed fruits or cereals, or of unwholesome food of any kind, is prohibited. The addition of all poisonous substances to food is prohibited, even when such poisonous substance is added in so small an amount as to be ordinarily innocuous. The addition of drugs to food is prohibited, except that materials ordinarily used as foods may be used by druggists for the purpose of preparing medicines in their ordinary forms. The use of injurious coloring materials is prohibited, both as a mixture with foods and in coloring wrappers. The use of poisonous metals, such as lead, zinc, tin containing more than 1 per cent of lead, and tin or copper containing more than 1 per cent of antimony is prohibited.

Tinned receptacles must not be coated with an alloy containing more than 1 per cent of lead or more than 0.01 per cent of arsenic. Glazed earthenware which is intended as a receptacle for food must not contain lead so combined as to be extracted by vinegar. Water used in the preparation of brandy and other alcoholic beverages must be pure, clear, and free from unwholesome ingredients. The use of injurious colors and aromatic essences in the manufacture of brandy is prohibited.

ALCOHOLIC BEVERAGES.

The alcohol used in the preparation of alcoholic beverages must contain none of the first or last distillates, must be free from acetic ether, fusel oil, and furfural. It must contain at least 95 per cent of ethyl alcohol and must answer to the following tests: 10 grams when treated with an equal weight of strong sulphuric acid remains colorless; 10 grams when treated with an equal weight of a solution of potassium hydroxid (specific gravity 1.3) must remain colorless; one volume when thoroughly mixed with five volumes of water must not be turbid or opalescent; from 20 to 25 cc when treated in a porcelain capsule with ten drops of colorless aniline or three drops of concentrated hydrochloric acid must remain colorless. The percentage of fusel oil present must not exceed 0.2 per cent of the absolute alcohol present; that of acetic ether must not exceed 0.02 per cent; that of furfural must not exceed 0.01 per cent.

Alcoholic beverages must not contain an excessive amount of aldehydes, furfural, methyl alcohol, or fusel oil. The addition of aniline derivatives and alkaloids of nitrobenzene, piperine, capsaicin, cocaine, ethyl nitrite, ethyl nitrate, ethyl ether, methyl ether, amyl ether, and the ethers of the various capronic and caprylic acids is prohibited.

Aloes, gamboge, jalap, or saccharin must not be added. The use of mineral acids and the compounds of the heavy metals such as lead, copper, and zinc is forbidden. The use of alum and of refuse animal charcoal which has not been purified is forbidden. Alcoholic beverages may be colored only with the following: Turmeric, alcoholic extract of carrots, safranin, safflower, marigold, cochineal, carmin, orseille, sandal red, Brazil wood, mallow, whortleberries, currants, raspberries, cherries, anchusa roots, indigo carmin, caramel, chlorophyll preparations, and litmus. For varying shades mixtures of the above may be employed.

The use of the following colors with alcoholic beverages is prohibited: Aniline colors of all descriptions; colors containing copper, lead, zinc, aluminum, antimony, and arsenic.

The addition of alcohol and the use of sulphurous acid for the purpose of regulating the fermentation in the preparation of distilled beverages is prohibited.

Distilled liquors must have the following alcohol content:

- Ordinary brandy from 12 to 35 per cent by volume;
- Plum brandy from 20 to 35 per cent by volume;
- Cherry brandy from 15 to 40 per cent by volume;
- Sweetened liqueurs, crèmes, rosolio, etc., from 15 to 40 per cent by volume;
- Cognac from 45 to 70 per cent by volume;
- Rum and arak from 45 to 70 per cent by volume.

WINE.

Wine is described as a product of the alcoholic fermentation of grape must, without addition of any description. If the source of the wine is not given it must answer the following description:

The extract content must not be less than 1.4 grams per 100 cc for white wines and 1.7 grams per 100 cc for red wines. Sweet wines and southern dessert wines must contain at least 3 grams of extract per 100 cc.

The minimum limit for ash content is one-tenth that of the extract, viz, 0.14 gram per 100 cc in white wines and 0.17 gram per 100 cc in red wines, while the ash content of southern sweet wines must not be less than 0.3 gram per 100 cc.

The percentage of alcohol must be between 6.5 and 15 per cent by volume. Southern sweet wines must contain from 8 to 20 per cent of alcohol by volume and sparkling wines from 8 to 15 per cent by volume.

The glycerin content must be at least 7 parts by weight for 100 parts of alcohol. Sweet wines must contain sugar in the proportion of 30 per cent for an alcohol content of 15 per cent.

The content of fixed acids must be at least 0.45 gram per 100 cc and the tartaric-acid content must be from one-fifth to one-sixth of the fixed acids present. The sodium-chlorid content must not exceed 0.005 gram per 100 cc and the sulphuric acid, calculated as potassium sulphate, must not exceed 0.2 gram per 100 cc.

Sparkling wines must not contain more than 0.05 gram potassium sulphate per 100 cc. Wines must not contain more than 0.0008 gram of free sulphurous acid or less than 0.0035 gram of phosphoric acid (P_2O_5), per 100 cc.

New wines whose fermentation is not completed must contain at least 1.55 gram extract per 100 cc, exclusive of sugar. Wines which

do not come within the standard given above or which contain more than 0.2 gram of acetic acid per 100 cc must not be sold as beverages. The sale of wine prepared from dried raisins and the addition to wine of any substance other than the product of the fresh grapes, except in the manufacture of medicinal preparations, is forbidden.

Wines made by the alcoholic fermentation of dry raisins, of mixtures of raisins with grapes, or of saccharine solutions of any sort other than pure musts, and those treated as follows are held to be adulterated:

The mixing with wines of organic or inorganic acids, or aromatic essences; the addition of glycerin, salicylic acid, boric acid, barium sulphate, strontium, aluminum and magnesium compounds, and of such substances as gum, dextrin, and resin, for the purpose of increasing the extract content.

The addition of the following substances to wine is especially prohibited:

Compounds of aluminum, magnesium, strontium, barium; the sulphites and sulphates of calcium and sodium; free mineral acids, compounds of lead, zinc, tin, copper, and arsenic; mineral colors and aniline colors of all descriptions; glucose, molasses, or crude sugar; crude alcohol; glycerin; boric acid and salicylic acid and their compounds; artificial essences and saccharin; pokeweed berries and juice of the same.

The following methods of treatment are permitted:

The blending of pure wines; neutralization of excessive acidity with calcium carbonate; filtration through pure vegetable charcoal; the use of clarifying agents, such as gelatin, albumin, isinglass, and kaolin; the sulphuring of empty casks by means of pure arsenic-free sulphur; the addition of pure refined spirits to sweet wine in such quantities that the limits given above shall be retained; the addition to sweet wine of refined sugar or glucose in such quantities that the limits given above shall be retained; the washing of casks with refined alcohol before they are filled, provided that the volume of the alcohol so employed does not exceed one-half per cent the volume of the wine manufactured; the addition of pure carbon dioxide in the preparation of carbonated wines; the plastering of red wines, provided that the product does not contain more than 0.2 gram potassium sulphate per 100 cc; the addition of must; and the pasteurizing of wines.

The manipulations mentioned above, however, must not be carried to such an extent that the composition of the wine will be rendered different from the required standards. All manipulations which change the character of the wine and serve to adulterate it are forbidden.

BEER.

Beer must be prepared exclusively from malted barley, hops, yeast, and water, by alcoholic fermentation. If a portion of the barley is replaced by any other material the product must be designated by a name indicating that fact.

Beer may vary in color from dark yellow to clear brown; it must have a characteristic odor and taste and be charged with carbon dioxide. It must contain from 2.5 to 6 per cent of alcohol, from 3.5 to 8 per

cent of extract, from 2.5 to 4.9 per cent of dextrin, and from 0.5 to 3 per cent of maltose.

The original wort from which it was prepared must have had an extract content of at least 9 per cent and the degree of fermentation must be at least 48 per cent. The total acid content must not exceed 0.35 per cent. The acetic-acid content must not exceed 0.06 per cent; the sulphuric-acid content must not exceed 0.2 per cent; the glycerin content must not exceed 0.04 per cent; the ash content must not exceed 0.3 per cent.

The addition to beer of alkaline carbonate for the purpose of neutralizing excessive acidity, of calcium or sodium sulphites, salicylic and boric acids, and similar compounds, is prohibited.

No coloring matter must be added except caramel and that naturally extracted from malt. The addition of saccharin, aromatic essences and extracts, hop substitutes, such as picric acid and its compounds, aloes, and all injurious substances in general, is prohibited.

VINEGAR.

Vinegar is defined as the product of the oxidation of refined alcohol or the fermentation of wine, beer, and the juices of various fruits, or as the mixture of pure concentrated acetic acid with pure water. It must be a clear liquid, free from suspended matter, and may have the color of the material from which it was prepared, varying from bright yellow to red, or it may be colored with pure caramel.

Vinegar must not contain more than 8 per cent of acetic acid nor less than 4 per cent. The manufacture of vinegar from crude alcohol is prohibited.

The addition of the following substances to vinegar is prohibited:

Sodium chlorid; mineral acids; bisulphites; poisonous metals and similar substances, such as lead, copper, zinc, arsenic, and antimony; black pepper, cayenne pepper, or other substances for the purpose of giving a sharp or bitter taste; products of the destructive distillation of wood (acetic acid excepted), such as methyl alcohol, acetone, etc.

CHEESE.

Cheese must contain nothing but the normal casein, proteids, butter fat, milk sugar, and mineral bodies obtained in its preparation from pure milk. Its reaction must be neutral or acid. The sale of cheese manufactured from milk of diseased or improperly fed cows is prohibited. The addition to cheese of any foreign substance, such as alkali, for the purpose of neutralization, foreign animal or vegetable fat, starch, and flour is prohibited. The addition of artificial essences for the purpose of giving a ripe taste to green cheese is prohibited. The addition of injurious colors and of artificial colors in general and of chemical preservatives is prohibited.

BUTTER.

Butter is defined as the product of milk or cream of the cow or buffalo. It is white or yellow in color, of uniform consistency, and contains a small amount of casein, milk sugar, lactic acid, unorganized bodies, etc. Butter must contain at least 82 per cent of fat, and the sale of butter prepared from adulterated milk or the milk of diseased or improperly fed cows is prohibited.

Butter must have the normal taste and odor and be free from rancidity, bitterness, fungi, dirt, etc. The addition of injurious artificial, mineral, or vegetable colors is prohibited. The content of sodium chlorid must not exceed 8 per cent, and the addition of foreign materials, such as starch, flour, and foreign fats is prohibited.

LARD AND TALLOW.

The addition to lard and tallow of foreign materials, such as foreign fat, alum, calcium carbonate, gypsum, sodium carbonate, starch, flour, and the sale of rancid and deteriorated fat are forbidden.

VEGETABLE OILS.

The sale as foods of vegetable oils obtained with the assistance of heat, hot water, steam, or by means of heating the press, or separated by means of such solvents as carbon disulphid, petroleum ether, and benzene, is prohibited. The admixture with olive oil of any other oil, such as sesame, peanut, rape-seed, sunflower, cotton-seed, mineral, and animal oils, is prohibited.

The sale as food of the oil prepared from decayed or fermented olives is prohibited. Table oil must be free from rancidity, and the total acid content must not exceed 1.66 per cent. The following are the requirements as to specific gravity of the oils mentioned:

Rape-seed oil, 0.914 to 0.917; olive oil, 0.915 to 0.918; oleo oil, 0.915 to 0.922; cotton-seed oil, 0.922 to 0.931; sesame oil, 0.923 to 0.924; poppy oil, 0.924 to 0.937; nut oil, 0.925 to 0.927; linseed oil, 0.932 to 0.937.

CEREALS AND FLOUR.

Cereals which are unripe, decayed, or decomposed, covered with fungus, affected by vegetable or animal parasites, or mixed with other varieties of cereals, can not be sold for human food, nor shall flour or meal prepared from the above be sold as food. The sale of a mixture of wheat, rye, barley, or maize flour with leguminous flour or other foreign material, except when properly designated, is forbidden. The ash of cereals and of the flour prepared from the same must have an alkaline reaction. The sale of flour which has deteriorated in any way or which contains more than 18 per cent of water is forbidden. The

sale of wheat flour which contains a mixture of the flour of any other substances, such as rye or barley, is forbidden. The addition of mineral substances, such as calcium carbonate and gypsum, is forbidden.

COFFEE, TEA, COCOA, AND CHOCOLATE.

The adulteration of coffee with any foreign substances, or of coffee from which any ingredient has been extracted, is prohibited. The mixture with coffee of artificial coffee beans or the sale of artificially colored coffee, or of coffee treated with any oil, roasted after the addition of sugar, or which has spoiled or deteriorated in any way, is prohibited. The sale of coffee substitutes may be permitted under some appropriate designation, such as "chicory," "barley coffee," and "fig coffee." These substitutes, however, must be free from injurious substances, and must not contain more than 5 per cent of ash or more than 14 per cent of moisture.

The term "cocoa" must be applied exclusively to the product of the cocoa bean. Cocoa powder, from which a portion of the fat has been removed, may be sold in packages which are so designated as to inform the purchaser of their nature, provided that they shall contain at least 22 per cent of cocoa butter. The term "soluble cocoa" may be applied to the same product when alkalized, provided that it contain not more than 2 per cent of potassium or sodium carbonate. The addition of artificial coloring matter, of foreign starch or fat, or foreign substances of any description, and the sale of cocoa so adulterated are prohibited. The sale of cocoa butter containing an excessive amount of cocoa shells is forbidden.

The term "chocolate" is applied to the product of the cocoa bean which has been mixed with sugar, with or without the addition of such flavoring materials as vanilla, cinnamon, etc. The sale of tea which contains the leaf of any other plant, which contains any foreign substance, or whose nature has been changed by extraction, is forbidden.

SUGAR, HONEY, CONFECTIONS, ETC.

It is forbidden to sell confections in receptacles of poisonous metals, or in receptacles which are tinned or coated with an alloy containing more than 1 per cent of lead, or which have in their composition any metal or glaze which is attacked by the confection or the sirup containing it.

Honey is defined as being the natural product of the bee, and containing from 78 to 92 per cent of invert sugar; from 1 to 3 per cent of cane sugar; from 1 to 2 per cent of proteids; from 0.12 to 0.44 per cent of ash; from 10 to 16.5 per cent of water.

Glucose which is intended for use in manufacturing confections must be commercially pure, and must contain from 88 to 95 per cent of

glucose; from 5 to 12 per cent of water; not more than 0.5 per cent of ash, and must contain no unfermentable matter, preservatives, or other foreign material.

Sugar must not be mixed with grape sugar, ultramarine, or indigo blue to a greater extent than 0.2 per cent; nor with gypsum, barites, kaolin, flour, saccharin, dulcin, or other similar impurities.

Confections must not be mixed with dulcin, with flour, mineral substances, or with those coloring matters which are prohibited in the general regulations regarding artificial colors. They must not be ornamented with flowers, leaves, etc., which contain injurious coloring material nor inclosed in receptacles or wrappers colored with injurious compounds.

The following colors are permitted:

White.—Ground cereal and potato flour.

Yellow.—Carrot, safranin, logwood, marigold.

Red.—Sorrel, madder, cochineal, carmine, red sandalwood.

Green.—Chlorophyl, spinach, and the mixtures of yellow and blue colors that are themselves permissible.

Blue.—Litmus and indigo carmine.

Violet.—Mixtures of blue and red colors that are permissible.

Brown.—Caramel, cocoa beans, licorice.

Black.—Purified bister.

The use of all colors which contain antimony, arsenic, barium compounds, cadmium, chromium, tin, copper, mercury, lead, uranium, zinc, picric acid, and aniline derivatives is prohibited. The use of gilded or silvered bronze or tinfoil which contains tin, lead, zinc, nickel, antimony, or aluminum, is forbidden.

SAUSAGE.

Sausage and other forms of preserved meat must be free from liver, kidneys, lungs, and viscera and consist entirely of the flesh of edible domestic animals, game, and birds put up while fresh.

The preparation of canned and preserved meat products from unsound or unwholesome meat or from the flesh of diseased animals or of other animals than those ordinarily used as food is forbidden. The preparation of canned and preserved fish which has been killed by means of poisonous substances, the manufacture of food products from the same, and the preserving of fish products in oil that is rancid or for any reason not edible, is prohibited. The use of commercial preservatives, such as salicylic acid or boric acid, tannin, alum, sulphurous acid, potassium chlorid, sulphites, glycerin, wood vinegar, impure vinegar, fusel oil, and other unwholesome substances for the preservation of meat or vegetables, is prohibited.

The coloration of preserved vegetables and fruits with mineral and aniline colors is forbidden, as is also the coloration of sausages and preserved meats.

TUNIS.

WINE

Wine is defined as the product of the fermentation of fresh grapes. The product obtained by the fermentation with water of the residuum of fresh grapes (after expression), whether with or without the addition of sugar, and the mixture of this product with wine in whatever proportion, can not be sold unless properly designated on all casks and receptacles and on all books, invoices, bills of lading, etc.

The product of the fermentation with water of dried raisins can not be sold except under the name of raisin wine. The same holds true in the case of mixtures of raisin wine with true wine, whatever may be the proportion.

Any addition of the following substances to wine is considered an adulteration:

1. Any coloring matter whatever.
2. Sulphuric, nitric, hydrochloric, salicylic, boric, or other analogous acids.
3. More than 0.1 gram of sodium chlorid per 100 cc.
4. The product of the fermentation or distillation of figs, locust pods, pimperl flowers, bellflower, rice, barley, and other materials containing sugar.

The casks or receptacles in which plastered wine is placed must be marked with large letters indicating the same. The books, bills of lading, invoices, etc., must contain the same information. The content of potassium sulphate must not exceed 0.2 gram per 100 cc in any case.

SWITZERLAND.

GENERAL PROVISIONS.

Beer must be made exclusively of cereals, either fresh or malted, hops, yeast, and water, by means of mashing and alcohol fermentation. All beer when sold must be clear and not rendered turbid by yeast, bacteria, acetic fermentation, or in any other manner. In the preparation of beer the following are prohibited: Malt and hop substitutes, all coloring matter except that of malt, preservatives such as salicylic acid and boric acid, and saccharin; and the addition of alkalies for the purpose of correcting excessive acidity.

Sulphurous acid must not be present in greater quantities than 0.0014 gram per 100 cc. Beer shall contain more extract than alcohol, and the extract content of the original wort must not be less than 12 per cent. The extract content of the wort is obtained by adding together the extract content of the beer and twice its alcohol content. The degree of fermentation must not be less than 48 per cent, or if less than that amount the reducing substances present, calculated as maltose, must not exceed 3 per cent. The degree of fermentation of the original wort is obtained by the formula $100 \left(1 - \frac{\text{Extract}}{x}\right)$, in which x is

the extract of the original wort. The foregoing standards do not apply to the so-called double beers, such as bock beer and salvator beer.

CANTON OF BERNE.

The addition to meat of boric acid, salicylic acid, formalin, sulphites, and all other chemical preservatives, except sodium chlorid and potassium nitrate, is prohibited.

CANTON OF GRAUBÜNDEN.

Meat.—Meat and meat products must have an appetizing appearance, a normal odor and taste, and must not contain any harmful impurities, such as metallic poison, drugs, ptomaines, parasites, etc. The addition of preservatives, with the exception of salt and saltpeter, is forbidden. Sausage must not contain more than 70 per cent of water, and bread crumbs, etc., shall not be added.

Butter and butter fats.—The term “butter” shall be used only with reference to the product of fresh milk and cream, either in the fresh state or the melted fat of the same. The fat content of fresh butter must be at least 82 per cent. Butter shall not form a part of the name of any product containing fat from other sources than pure milk. The sale as food of fat which has become rancid, or has in any way deteriorated, is forbidden.

Flour and meal.—All flour and meal must be so marked as to indicate the grain from which it is prepared. It must be free from mineral impurities, fungi, and weed seeds.

Canned vegetables.—Canned vegetables must not contain over 10 mg of copper salts per 100 grams of fresh food.

Honey.—The term “honey” must be confined to the unmixed product of the bee. It shall not be used either by itself or in combination with other syllables or words to designate adulterated honey or honey substitutes. Such adulterated honey and honey substitutes must be inclosed in receptacles bearing labels on which the term “sirup” appears in distinct type. Also all invoices and shipping receipts of such adulterated goods must be marked with the word “sirup.”

Beer.—The term “beer” must be used only in reference to beer made exclusively from malted barley, hops, yeast, and water, by means of mashing and alcoholic fermentation. In case part of the barley is replaced by some other cereal the same must be plainly stated on the label. Malt and hop substitutes are prohibited. Beer must be clear, wholesome, and free from yeast; the original wort from which it was prepared must have had an extract content of at least 12 per cent. Beers whose degree of fermentation is less than 48 per cent must not contain over 3 per cent of maltose. These regulations do not apply to the so-called double beers, such as bock beer and salvator beer.

The ash content must not exceed 0.3 per cent, and the sulphurous acid content must not exceed 0.004 gram per 100 grams. The presence of boric and salicylic acids in beers is forbidden.

Wines.—The term “wine” shall be applied exclusively to the beverage prepared from the juice of fresh grapes without the addition of

any foreign substances. Wines whose volume has been increased by the addition of any foreign substances, or which are prepared from any other fruits than wine grapes, shall be so labeled as to indicate that fact. The sale of wines which have become sour or deteriorated in any way is forbidden. Wine whose sulphurous acid content, calculated as potassium sulphate, exceeds 0.1 gram per 100 cc shall be designated as "plastered;" if it exceed 0.2 gram per 100 cc it shall be designated as "excessively plastered." Wine must not contain more than 0.002 gram of free sulphurous acid or 0.018 gram of combined sulphurous acid per liter. A higher content of sulphurous acid is considered unwholesome. The addition of preservatives, such as boric and salicylic acids, is prohibited.

The alcohol content of medicinal wines shall not be less than 13 or more than 20 per cent by volume. They shall not contain less than 0.2 gram of ash or more than 0.2 gram of acetic acid, 0.2 gram of potassium sulphate, or 0.002 gram of total sulphurous acid, per 100 cc.

Brandy and liqueurs.—The presence of poisonous metallic compounds, such as copper or lead, and of free mineral acids is prohibited. The alcohol of brandy must not contain more than 0.2 per cent of fusel oil.

Vinegar.—Vinegar must not contain less than 3 per cent of anhydrous acetic acid. The presence of free mineral acid is prohibited. The sale as wine vinegar of vinegar made from any other substance than wine is prohibited.

Receptacles.—All receptacles and wrappers for food must be free from harmful substances. The use of lead foil or of tin foil containing lead is especially prohibited.

Coloring matter.—The addition of artificial colors to meat or meat products, wines and similar beverages, beer, distilled and wood vinegar, coffee, tea, chocolate, condiments, fruit juices, fruit lemonades, and bakers' products supposed to contain eggs is prohibited. The addition to foods of artificial colors which contain harmful substances, such as the following, is prohibited: Antimony, arsenic, barium, lead, cadmium, copper (except that copper salts may be added to canned vegetables in amounts not exceeding 10 mg per 100 grams), chromium, mercury, zinc, and tin. The use of gamboge and injurious aniline colors is also prohibited.

CANTON OF LUCERNE.

The adulteration of foods by extracting from, adding to, or changing in any way that will decrease the value, is prohibited. Only substances may be added which are necessary in preparation, transportation, or preservation, and which do not increase weight or injure quality. The name must not misrepresent place and manner of pro-

duction and manufacture. Food that is unripe, unsound, or for any reason unfit for food must not be sold. Standards for cacao, etc., vinegar, honey, coffee, flour (wheat or rye), milk, must, tea, drinking water, and wine are given.

Beer.—Beer must contain more extract than alcohol, and must be prepared from wort containing not less than 12 per cent of solids. The glycerine content must not exceed 0.4 per cent. Not more than 3 cc of normal alkali shall be required for the neutralization of total acids in 100 grams of beer from which carbon dioxid has been removed by shaking. Not more than 1 cc of normal soda solution shall be required for the neutralization of volatile acids. The content of sulphurous acid must not exceed 0.0014 grams per 100 cc. At least 48 per cent of the original extract of the wort must have been fermented. These standards do not apply to the so-called double beers (bock beer and salvator). Beer which is turbid because of the presence of yeast or bacteria shall not be sold. The addition of unwholesome preservatives, such as calcium bisulphite, and of alkaline substances, such as potash and soda, for the purpose of correcting excessive acidity, is prohibited. The use of so-called beer color (caramel, etc.) is prohibited.

Brandy.—The addition of 15 cc of brandy to an equal volume of distilled water and a few drops of a solution of potassium ferrocyanide should not produce a red-brown precipitate, and the addition of an excess of ammonia must not cause a marked blue color (presence of copper). Brandy must contain no trace of lead or free inorganic acid. The content of fusel oil must not be sufficient to produce a turbidity when the brandy is mixed with 3 volumes of water, or to allow the globules of fusel oil to separate when 1 volume of brandy is mixed with 1 volume of ether and 2 volumes of water. Brandy must contain at least 46 per cent of alcohol by volume, except old brandy, whose alcoholic content may be as low as 44 per cent by volume.

Butter.—Butter must contain no fat except that prepared from milk. Fresh butter must contain at least 82 per cent of butter fat. The word "butter" must not be used, even in combination with other words, to designate articles containing fat from other sources than milk. For instance, such terms as "Kunstbutter" (artificial butter) and "Kübelbutter" (tub butter) are not permitted for articles containing fat from other sources than milk.

Cocoa and cocoa preparations.—Foreign additions to cocoa, such as flour, starch, and spices, and even sugar, must be stated on the outside of each package. The addition of alkaline carbonate not to exceed 2 per cent may be made to the hulled cocoa powder for the purpose of rendering it soluble.

Vinegar.—Vinegar must contain at least 4 per cent of acetic acid. The addition of other acids, of pungent or aromatic substances, and

of aniline colors, is forbidden. Vinegar made by diluting so-called vinegar essence must be designated as "essence vinegar."

Honey.—Only the unsophisticated product of bees can be designated as honey. The word honey can not be used in combination with other words to designate any article other than pure bee honey; for instance, such terms as "table honey" and "Swiss honey" are permitted only for pure honey. All honey substitutes, such as commercial glucose, molasses, and all mixtures of the same with honey, must be so labeled as to inform the purchaser of the exact origin and composition of the contents of the package.

Coffee.—Coffee must not contain more than 4 per cent of ash, except Mocha coffee, which may contain 8 per cent. The use of artificial colors in coffee and the fraudulent mixture of adulterants is prohibited.

Flour.—The ash content shall not exceed 2 per cent for rye flour or $1\frac{1}{2}$ per cent for wheat flour. The water content of wheat and rye flour must not exceed 15 per cent. That of other varieties must not exceed 18 per cent.

Cider.—Fermented cider shall not contain less than 3 per cent of alcohol by volume, 1.5 per cent of extract, or 0.15 per cent of ash.

Wine.—Wine must not contain less than 6.24 per cent of alcohol by volume. The extract content must not be less than 1.5 per cent for red wine or less than 1.4 per cent in white wine. The ash content of wine must be at least 0.15 per cent. By ash content is meant carbonated ash. The percentage of volatile acid expressed as acetic acid must not exceed 0.2 per cent. The sulphuric acid (combined) content, expressed in terms of potassium sulphate, must not exceed 0.2 per cent for medicinal wine and must not exceed 0.1 per cent in dry wines. Wines which contain over 0.008 grams of sulphurous acid per 100 cc must not be sold for consumption without previous cellar manipulation. These restrictions are not applied to sweet or sparkling wines, and apply only to medicinal wines when the latter are specified. The addition of artificial colors is prohibited.

Sausage.—Sausage and any similar preparations must have been prepared exclusively from sound, fresh meat, fat, liver, and blood, with the customary addition of spices. All other additions, such as starchy materials, are considered as adulterants.

CANTON OF ST. GALLS.

All materials intended for food must be so labeled as to inform the purchaser as to their exact nature. The sale of adulterated or unwholesome foods is prohibited. The usual regulations concerning the sale of butter, oleomargarine, lard, etc., are enforced. The so-called St. Gall's sausage (Kalbfleischbratwurst) must not contain more than 2 per cent of added starch or flour. Horse-meat sausage must not contain more than 3 per cent of starch or flour. The sale of cider and

similar preparations made from green fruit is forbidden. The sale of all foods contaminated with poisonous metals, such as zinc and lead, or inclosed in receptacles lined with zinc or lead alloys, is forbidden.

Wine.—Medicinal wines must not contain more than 0.002 gram of total sulphurous acid, more than 0.2 gram of sulphuric acid expressed in terms of potassium sulphate, or more than 0.2 gram of acetic acid per 100 cc. White wines must not contain more than 0.002 gram of free sulphurous acid or more than 0.018 gram of combined sulphurous acid per 100 cc. Wine whose sulphurous-acid content exceeds this limit is considered unwholesome and must be subjected to cellar manipulation before it is sold as a beverage. Wine which has become sour or turbid owing to the acetic-acid fermentation, or which has deteriorated in any other way, must not be sold as a beverage.

Beer.—The addition of alkaline substances for the purpose of neutralizing the excessive acidity of beer is prohibited. The addition to beer of salicylic or boric acid is prohibited. The sulphurous-acid content of beer must not exceed 0.0014 gram per 100 cc. New beer or beer which has become turbid by reason of the presence of yeast cells or bacteria must not be sold as a beverage.

CANTON OF ZURICH.

The addition of all preservatives to meat, except salt and saltpeter, is prohibited.

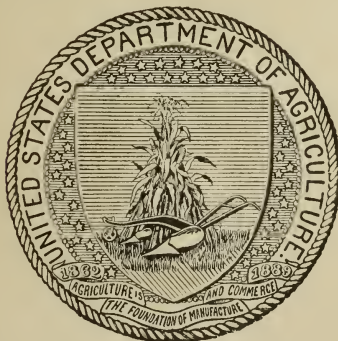
Coffee substitutes shall be named according to the chief ingredient when possible, as "chicory coffee," "malt coffee," etc. When the product is a mixture of a number of substances, it shall be designated as "Kaffee-surrogate," and either the chief constituents shall be printed on the label or all of the constituents communicated to the board of health.

U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY.

PROCEEDINGS
OF THE
SEVENTEENTH ANNUAL CONVENTION
OF THE
ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS
HELD AT
WASHINGTON, D. C.

NOVEMBER 16, 17, AND 19, 1900.

EDITED BY
HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.



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1901.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,

Washington, D. C., January 28, 1901.

SIR: I have the honor to transmit to you herewith the manuscript of the proceedings of the seventeenth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 62 of the Division of Chemistry.

H. W. WILEY,

*Chief of the Division of Chemistry and Secretary of the
Association of Official Agricultural Chemists.*

Hon. JAMES WILSON,

Secretary of Agriculture.

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PROCEEDINGS OF THE SEVENTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

FRIDAY—MORNING SESSION.

The seventeenth annual convention of the Association of Official Agricultural Chemists was held in Washington, D. C., beginning November 16, 1900. The meeting was called to order by the president, Mr. B. W. Kilgore, in the lecture hall of the Columbian University.

MEMBERS AND VISITORS PRESENT.

The following members and visitors were in attendance:

Allen, W. M., Assistant State Chemist, Raleigh, N. C.
Anderson, J. T., Agricultural Experiment Station, Auburn, Ala.
Bache, A. W., U. S. Department of Agriculture, Washington, D. C.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Beal, W. H., U. S. Department of Agriculture, Washington, D. C.
Bergami, Francis, Chemist, Baugh & Sons Co., Philadelphia, Pa.
Bigelow, W. D., U. S. Department of Agriculture, Washington, D. C.
Brackett, R. N., Agricultural Experiment Station, Clemson College, S. C.
Cameron, F. K., U. S. Department of Agriculture, Washington, D. C.
Carpenter, B. F., Chemist, Virginia-Carolina Chemical Company, Richmond, Va.
Carr, Oma, care J. P. Heald & Co., Lynchburg, Va.
Church, C. G., Maryland Agricultural College, College Park, Md.
Covert, J. R., U. S. Department of Agriculture, Washington, D. C.
Davidson, R. J., Agricultural Experiment Station, Blacksburg, Va.
Dyer, B., London, England.
Edelen, George S., Maryland Agricultural College, College Park, Md.
Ewell, E. E., U. S. Department of Agriculture, Washington, D. C.
Fields, John, Agricultural Experiment Station, Stillwater, Okla.
Fraps, G. S., Agricultural Experiment Station, Raleigh, N. C.
Frear, William, Agricultural Experiment Station, State College, Pa.
Gough, T. R., Maryland Agricultural College, College Park, Md.
Hand, W. F., Mississippi Agricultural College, Agricultural College, Miss.
Hardy, J. C., Mississippi Agricultural College, Agricultural College, Miss.
Herff, B. von, German Kali Works, New York, N. Y.
Hills, J. L., Agricultural Experiment Station, Burlington, Vt.
Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
Holland, E. B., Hatch Experiment Station, Amherst, Mass.

Huston, H. A., Agricultural Experiment Station, Lafayette, Ind.
 Jones, C. H., Agricultural Experiment Station, Burlington, Vt.
 Kilgore, B. W., State Chemist, Raleigh, N. C.
 Krug, W. H., U. S. Department of Agriculture, Washington, D. C.
 Lamb, F. C., Raleigh, N. C.
 Langworthy, C. F., U. S. Department of Agriculture, Washington, D. C.
 Laughlin, J. L., Maryland Agricultural College, College Park, Md.
 Le Clerc, J. A., Agricultural Experiment Station, Geneva, N. Y.
 Leech, A. E., State Board of Health, Boston, Mass.
 McCandless, J. M., State Chemist, Atlanta, Ga.
 McDonnell, H. B., Agricultural College, College Park, Md.
 McMurtrie, William, 100 William street, New York.
 Macfarlane, T., Ottawa, Canada.
 Magruder, E. W., Richmond, Va.
 Mattern, L. W., Central High School, Washington, D. C.
 May, D. W., U. S. Department of Agriculture, Washington, D. C.
 Meng, James S., German Kali Works, New York, N. Y.
 Miller, H. K., Agricultural Experiment Station, Lake City, Fla.
 Mills, J. S., Washington, D. C.
 Moore, C. C., U. S. Department of Agriculture, Washington, D. C.
 Moore, J. F., Agricultural Experiment Station, Fayetteville, Ark.
 Myers, J. A., 12 John street, New York, N. Y.
 North, J., care Eimer & Amend, New York, N. Y.
 Orton, W. A., U. S. Department of Agriculture, Washington, D. C.
 Patrick, G. E., U. S. Department of Agriculture, Washington, D. C.
 Patterson, B. C., Torrington, Conn.
 Patterson, H. J., Agricultural Experiment Station, College Park, Md.
 Penny, C. L., Agricultural Experiment Station, Newark, Del.
 Perkins, W. R., Agricultural Experiment Station, Agricultural College, Miss.
 Plunkett, W., 12 John street, New York, N. Y.
 Price, T. M., Agricultural Experiment Station, College Park, Md.
 Rand, Stephen, Washington, D. C.
 Robb, J. B., Maryland Agricultural College, College Park, Md.
 Robertson, A., Laidlaw Meekill Company, Richmond, Va.
 Runyan, E. G., U. S. Department of Agriculture, Washington, D. C.
 Sawyer, H. E., 620 Atlantic avenue, Boston, Mass.
 Schlichting, E., U. S. Department of Agriculture, Washington, D. C.
 Seidell, Atherton, U. S. Department of Agriculture, Washington, D. C.
 Shiver, F. S., Agricultural Experiment Station, Clemson College, S. C.
 Shutt, F., Ottawa Experiment Station, Ottawa, Canada.
 Shuttleworth, A. E., Ontario Agricultural College, Guelph, Canada.
 Smith, G. W., 75 West 45th street, New York, N. Y.
 Spencer, G. L., U. S. Department of Agriculture, Washington, D. C.
 Straughn, M. N., Maryland Agricultural College, College Park, Md.
 Street, J. P., Agricultural Experiment Station, New Brunswick, N. J.
 Stubbs, J. E., Agricultural Experiment Station, Reno, Nev.
 Tillinghast, J. A., Agricultural Experiment Station, Kingston, R. I.
 Tolman, L. M., U. S. Department of Agriculture, Washington, D. C.
 Traphagen, F. W., Agricultural Experiment Station, Bozeman, Mont.
 Trescot, T. C., U. S. Department of Agriculture, Washington, D. C.
 Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
 Veitch, F. P., U. S. Department of Agriculture, Washington, D. C.
 Voorhees, L. A., Agricultural Experiment Station, New Brunswick, N. J.
 Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.

Wiley, H. W., U. S. Department of Agriculture, Washington, D. C.
 Williams, C. B., Department of Agriculture, Raleigh, N. C.
 Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
 Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.

ORDER OF BUSINESS.

The following order of business was adopted:

The president's address.

Reports of the referees in the following order:

First. Report on nitrogen.

Second. Report on potash.

Third. Report on phosphoric acid.

Fourth. Report on soils.

Fifth. Report on ash.

Sixth. Report on foods and feeding stuffs.

Seventh. Report on liquor and food adulteration.

Eighth. Report on dairy products.

Ninth. Report on sugar.

Tenth. Report on tannin.

Eleventh. Report on insecticides.

Twelfth. Reports of special committees (abstract committee, food standards, fertilizer legislation, volumetric standards).

The president announced to the association that a representative of the National Grange was present and would address the members. He then introduced Mr. J. A. Tillinghast, of Kingston, R. I., who stated that he had come to invite the members of the Association of Official Agricultural Chemists to meet in connection with the National Grange at 11 o'clock at the National Hotel.

It was moved and carried that after the reading of the president's address the association should adjourn to meet with the National Grange at the National Hotel, and that it should reassemble in the lecture hall of the Columbian University at 1 o'clock p. m.

The president appointed Mr. Frear and Mr. Van Slyke a committee to wait upon the Secretary and Assistant Secretary of Agriculture and notify them that the members of the association would be pleased to have them visit the meeting.

Mr. Kilgore then addressed the convention as follows:

PRESIDENT'S ADDRESS.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: In casting about for a subject on which to address some remarks to those attending the seventeenth annual convention of the association over which you have done me the great honor, which I highly appreciate, of calling me to preside, I have read with interest and profit all of the accessible former presidential addresses before

this body and have found that without enactment on the part of the association, custom of sixteen years' standing has practically fixed the basis of the presiding officer's remarks, unless he essays to be a reformer. Without exception, these addresses have been short and have dealt with some phase of our history as an association, with present work or problems confronting the agricultural chemist, with specific discussion relating to the improvement of some of our methods for the analysis of agricultural products, or with suggestions for enlarging the field of our endeavor.

The records of the association show it to be one of action and business, and I shall endeavor to put what I have to say before you in a business way.

NATIONAL PURE-FOOD LAW.

Not only has the scope of our work been extended, but the influence of the association has grown. During the year we have been called upon as an organization to aid in the education of public sentiment and to bring influence to bear in favor of a national pure-food bill, with which we are all more or less familiar. Knowing that a number of the members of the association are actively engaged in the examination of food products and in aiding to execute State pure-food laws, and feeling that our entire membership is deeply interested in this important movement upon which so much of health and happiness for ourselves and those we represent depends, I used the name of the association and joined other scientific bodies in this country in asking our Congress to pass a national pure-food law. I trust that I may have the approval of the association in this action.

In North Carolina we are just preparing our first report on the examination of food products under our new pure-food law. We have been engaged in this work, as time permitted, for over a year and see the importance and necessity for a national law to help us when we can not help ourselves. As other scientific bodies have adopted resolutions on this subject, and in view of its importance, I suggest that this association, possibly through resolutions prepared by its standing committee on food standards, express itself fully in regard to the matter.

NATIONAL STANDARDIZING BUREAU.

Accurate and uniform standards are the basis of analytical and, in fact, all scientific work. Especially are they essential to the work of this association, the fundamental feature of whose organization is uniform methods as a means to uniform results. In the last Congress the prospects seemed fair for the passage of a bill establishing "The National Standardizing Bureau" under Government control. It would be the work of this bureau to investigate and adopt standards,

and to provide facilities for testing and correcting the apparatus of various scientific and other workers, and to establish uniform systems. Since 1896 this association has had a standing committee on volumetric standards, and in the discussion and approval of the three reports made by this committee the association has shown its deep interest in the question of accurate and uniform standards. With this in view, I again used the name of the association in trying to help secure the passage of the bill to which reference has already been made.

I can not think of anything that is more essential to the progress of the work of this association than that involved in the question of proper standards; and, if I mistake not, it was here that this movement had its beginning in this country. The question was up for discussion in the 1896 meeting, when provision for a committee was made. That committee has made three reports of the results of its investigations, and a member of the committee brought the matter before the American Chemical Society, which is now active in its support of the measure. Our association and its members have, therefore, done their part of the work on this important question. Should we not now express fully and clearly our views on the subject of standards so that they may be used with the full force of the association in case the matter comes up again for agitation?

UNIFORM STATEMENT OF RESULTS OF FERTILIZER ANALYSES.

In addition to obtaining uniform, accurate, rapid, and inexpensive methods of analysis, this association has among its objects the securing of uniform modes of statement of results obtained by these methods. We have paid more attention in the past to getting proper methods, which was certainly the first and most important object to be attained, than to securing uniform statements of the results. The work of the committee appointed in 1897, and which reported in 1898, on the subject of uniform branding of fertilizer bags and uniform fertilizer laws, has done good in that it has been made the basis of an agreement and report of the State chemists of seven of the Southern States to the Association of Commissioners of Agriculture of the Cotton States. This association met in Raleigh, N. C., during the past summer, and after a great deal of discussion the chemists' recommendations were agreed to, and we feel that there is now a hopeful outlook for the plan going into execution. It is necessary for the chemists and commissioners of agriculture, or others having the business of the fertilizer controls in charge, to come together in these matters, and such has been our mode of attack in the South.

Not only was an agreement reached in this meeting as to the statement of analyses on fertilizers, but the committee was continued and enlarged and instructed to draft a uniform fertilizer law for the States represented in the association, and also to meet with the manufacturers

if desirable and see if an agreement can not be reached in regard to the naming or misnaming of fertilizer brands, such, for instance, as "dissolved bone" and "dissolved bone and potash," when there is no bone present; and, further, to consider the character of materials entering into the different fertilizer brands, and to investigate the desirability of exacting the filing of a statement in regard to these, as is done in the case of the valuable constituents of a fertilizer.

I make this somewhat detailed statement to show the association that its actions have weight, and in the hope of inducing further discussion of this question in case the association has the time and inclination to do so. It may not at first thought seem so important to have uniformity in the naming of the valuable constituents of fertilizers, and so far as chemists alone are concerned it is not, for we understand the various synonymous terms employed in designating them, but when it is remembered that the burden of explaining these terms to the farmers and users of fertilizers devolves upon the chemist, the question of sameness and simplicity assumes weight, and it would be greatly to our advantage and credit if we called the same substances by the same names in all of the States of the Union. This association can bring this about, as it has in the case of methods, if it will only put its shoulder to the wheel, and we will then have accomplished another object set forth in our constitution.

INDIVIDUAL EFFORT AS A FACTOR IN THE GROWTH OF THE ASSOCIATION.

I now desire to make a few observations on facts as they have impressed themselves upon me in a study of the history of the growth of this association. The most prominent subject before the American public to-day is that of trusts, combinations, and organizations. So prominent have these become that it is generally assumed that the individual is swallowed up and lost sight of in the great aggregations. Is this really true? Certainly in union there is strength, but we have strength and influence, not only because of our organization but because care, conservatism, and strong individual efforts have directed the various phases of our endeavors in which much progress has been made. I feel that I am correct in the observation that no very important advance in the development of any of the methods of the association has been accomplished except where strong individual effort has preceded the submission of the questions to the general test of the association. Those who are familiar with the work of the association for a number of years know that with each of the subjects which have been under investigation are associated the names of a few of the workers of the association who have become prominently identified with the perfection of each particular process or method only because of having given a great deal of time, thought, and labor to the solution of the problems before them. It is not true that they

deserve all the credit, nor is it right that they should receive it, for unless the association take up and subject their plans to the rigid test to which it submits everything, their ideas could not receive the acceptance and prominence which have been accorded them. Those men, however, because of their individual labors, deserve to be called captains of their causes.

We might trace the development of any of the older methods of the association, especially those employed in fertilizer analysis, to see how much is due to individual efforts in bringing any one of them to its present state of accuracy. The work on potash methods lends itself well to a study of this kind, and I shall refer briefly to the important steps in perfecting our methods for this determination.

The rather crude condition of the method generally used in 1880 and prior thereto may be imagined from the following description: "Solution in water; removal of sulphates, phosphates, and magnesia by barium hydrate; clearing with oxalate or carbonate of ammonia, and precipitating with platinum chlorid." The brevity of description was doubtless compensated for by the variation in manipulation. This method was used until 1884, when it was redescribed and adopted as the official method in such form as to require five filtrations, three evaporations, and two ignitions. By investigations conducted in 1885 one each of the filtrations, evaporations, and ignitions were omitted and the method of Lindo was given a test. This method, modified on the basis of the work and suggestions of Gladding, became one of our methods for potash determinations in 1886. As then described, only two changes need to be made to give us our most excellent Lindo-Gladding method of to-day. No change was made in this method from 1886 to 1888, when, on the basis of work done, especially at the New Jersey Experiment Station, it was directed to add ammonium oxalate to help remove lime. While a good many workers were now doubting the necessity of using sodium chlorid in this method, it remained for Winton to show, by a most carefully conducted series of experiments, the results of which were presented in a paper before the association in 1891, that it was not only not essential but was a hindrance and a great expense. This conclusion was verified by the general test of the association in 1892, when the term "sodium chlorid" was stricken from its place in the method.

Criticism, whether of individual work or of an association's position, is one of the strongest incentives to a thorough refutation of charges and the establishment of the correctness of the work or opinion beyond peradventure. Such criticism fortunately came upon our potash methods about 1893. Spurred on by the sting of unfavorable opinion, and under the wise and fortunate action of this body, which permits the long association of men with particular lines of work, our potash methods were subjected through the years 1894, 1895, 1896, and 1897

to such continuous, severe, and carefully planned and conducted tests on all classes of material that the results obtained must command the greatest respect and confidence of others, especially for our Lindo-Gladding method, and raise it in the estimation of its friends at home, where it was already most favorably known. While the association is to be congratulated and given credit as a whole for this excellent work, I feel that the greater part of the credit belongs to the two or three referees and associates whose individual efforts planned so well this continuous work and who verified personally the wisdom of their plans before submitting them to the general test of the association. To this I would add that we were greatly aided through the efforts of one of our members in securing this uniformly high opinion of our methods, by a better knowledge of the crystalline character of the end compound, the double chlorid of potassium and platinum, and of the conditions for obtaining it in a state best suited to accurate results.

So well has this work been done that the referee in 1896, in referring to the Lindo-Gladding method, said, "An experienced analyst, who follows strictly the instructions and uses a Gooch crucible, should find it rapid, economical, and accurate," while the referee for 1898 opened his report with these words: "The methods for estimating potash have been quite thoroughly studied by this association in years past, so far as potash salts and fertilizers are concerned," and he proceeded to test the applicability of the methods to different kinds of ashes. And, as further evidence of the satisfactory condition of our potash methods, it may be said that so excellent and ingenious a worker as Professor Ross could only suggest for investigation in 1899 the following question: "Which is better with which to saturate in making ignitions of such organic materials of cotton seed meal and tobacco stems, concentrated or 1:1 sulphuric acid?"

What I have said in regard to individual effort in connection with our work has been said without desire to detract from the great and supreme importance of the association, or to exalt unduly the work of individual members. I have merely stated some facts as they have impressed me in the study of our growth in the hope that they may induce more of our members to take up and study thoroughly some of the many phases of the subjects on which we are working and which are in such sore need of strong individual effort.

METHODS FOR THE EXAMINATION OF HUMAN FOODS.

In conclusion may I in a few words draw your attention to the growing prominence and importance of the examination of human food and drink. Recently a number of States have passed pure-food laws. We are in need of new methods and of concerted tests of old ones for the examination of food products. Our methods of fertilizer

analysis are in very satisfactory condition, and this status has only been reached by dividing the subject into nitrogen, phosphoric acid, and potash, and giving to each detailed consideration. The food analyst is called upon to make not only the usual analysis of the various foods, condiments, and drinks, but also to examine them for adulterants, preservatives, and coloring matters. Would it not be well for the association to make a number of subdivisions of this subject, and place experienced men in charge of the investigation of each subdivision? The scope of our work would be somewhat enlarged by an action of this kind and the food chemists generally would no doubt attend our meetings more largely in the future than they have in the past if the work of the association were thus made of more interest and value to them.

APPOINTMENT OF COMMITTEES.

At the conclusion of the president's remarks he declared the seventeenth annual meeting of the Association of Official Agricultural Chemists open for business, and appointed committees as follows:

Committee on recommendations of referees: H. A. Huston, J. T. Anderson, C. H. Jones, F. W. Traphagen and the referee of the report under consideration.

Committee on nominations: A. L. Winton, J. P. Street, W. F. Hand.

Committee on resolutions: C. L. Penny, F. T. Shutt, H. K. Miller.

Committee on subdividing work on liquor and food adulteration: A. L. Winton, H. W. Wiley, A. E. Leech, William Frear, W. D. Bigelow.

The meeting was then adjourned in order that the members of the association might attend, in a body, the meeting of the National Grange.

FRIDAY—AFTERNOON SESSION.

President Kilgore called the meeting to order at 1 p. m.

The referee on nitrogen not being present, the report on potash was called for and was presented by the secretary as follows:

REPORT ON POTASH.

By L. S. MUNSON, *Referee*, and C. L. HARE, *Associate Referee*.

The potash work of the association for several years past has been confined largely to investigations of analytical methods. This year it was thought desirable to include in the work the investigation of such problems as have presented themselves in connection with the application of the present methods to fertilizer work. Accordingly, on July 16, three samples were prepared and sent out to 19 persons

who had expressed a desire to cooperate in the work. Of these, 8 have reported results obtained by 11 different analysts, 6 have reported inability to do the work, owing to lack of time, and 5 have made no report whatever.

Sample No. 1 was cotton-seed meal.

Sample No. 2 consisted of a mixture of 162 parts of acid phosphate, 64 parts of ground tobacco stems, 10 parts of potassium chlorid, and 4 parts of magnesium sulphate.

Sample No. 3 was of 2 parts, No. 3A being acid phosphate and No. 3B chemically pure KCl.

The instructions sent out with these samples were as follows:

- (1) Determine moisture in samples Nos. 1 and 2.
- (2) Determine both water soluble and total potash in samples Nos. 1 and 2.
- (3) Make mixtures of No. 3A and 3B as follows:
 - (a) Nine grams acid phosphate and 1 gram chemically pure KCl.
 - (b) Nine and five-tenths grams acid phosphate and 0.5 gram chemically pure KCl.
- (4) Make blank determinations of potash in acid phosphate used in No. 3 and in reagents and determine potash in chemically pure KCl.
- (5) Make all determinations according to official methods.

Samples Nos. 1 and 2 were for the purpose of testing the application of the method for determining water-soluble potash to such fertilizers as are made up entirely or in part of organic material. The object of sample No. 3 was to ascertain whether in mixtures of acid phosphate and potash salts it was possible to obtain the theoretical amount of potash added.

The following results were obtained:

TABLE I.—*Comparison between water soluble and total potash in samples Nos. 1 and 2.*

Name.	Address.	Sample No. 1.			Sample No. 2.		
		Moisture.	Water soluble K ₂ O.	Total K ₂ O.	Moisture.	Water soluble K ₂ O.	Total K ₂ O.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
B. F. Robertson	Clemson College, S. C.	9.17	1.68	1.80	6.44	5.20	5.46
C. C. McDonnell	do.....	9.11	1.63	1.77	6.39	5.16	5.49
C. L. Hare	Auburn, Ala.....	7.65	1.79	1.95	6.60	5.37	
J. Q. Burton	do.....		1.84	1.92		5.40	5.46
A. W. Blair	Lake City, Fla.....	9.97	1.58	1.79	8.15	5.22	5.34
L. S. Munson.....	Washington, D. C....	8.24	1.78	1.89	6.54	5.28	5.38
C. D. Holley	Orono, Me	9.64	1.58	1.75	6.50	5.03	5.45
F. B. Carpenter	Richmond, Va.....		1.51	1.75		5.06	5.33
S. H. Sheib.....	do.....	9.14	1.37	1.75	6.38	5.07	5.26
T. R. Gough.....	Agricultural College, Md.	7.53	1.65	1.22	6.39	5.19	
I. D. Sessions.....	Agricultural College, Miss.	8.26	1.44		6.26	5.12	
Average		8.75	1.62	1.82	6.63	5.19	5.40

¹ Not included in average.

TABLE II.—*Results of analysis of mixture of acid phosphate and potassium chlorid.*

Name.	Address.	K ₂ O in C. P. KCl.	K ₂ O in acid phos- phate.	K ₂ O in mix- ture of 9 gr. acid phosphate and 1 gr. KCl.	K ₂ O in mix- ture of 9.5 gr. acid phosphate and 0.5 gr. KCl.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
B. F. Robertson	Clemson College, S. C.	62.62	0.04	6.01	2.94
C. C. McDonnell	do	62.61	.03	5.99	2.95
C. L. Hare	Auburn, Ala.	63.20		6.13	3.11
J. Q. Burton	do			6.20	3.16
A. W. Blair	Lake City, Fla.	62.96	.103	6.14	3.08
L. S. Munson	Washington, D. C.	63.08	.155	6.21	3.14
C. D. Holley	Orono, Me	63.00		6.30	3.15
F. B. Carpenter	Richmond, Va.		.06	6.19	3.10
S. H. Sheib.	do	63.13	.08	6.09	3.09
T. R. Gough	Agricultural College, Md.	63.30	.14	6.13	3.08
I. D. Sessions	Agricultural College, Miss.	160.65		5.89	2.92
Average		62.99	.083	6.12	3.07

¹Not included in average.

Theoretical per cent of K₂O in mixture of 9 gr. acid phosphate and 1 gr. KCl 6.37
 Theoretical per cent of K₂O in mixture of 9.5 gr. acid phosphate and 0.5 gr. KCl 3.23

From Table No. 1 it will be seen that the results obtained for water-soluble potash are extremely irregular. With sample No. 1 there is a variation of 0.47 per cent, with less than 2 per cent of potash present, and while the results obtained on sample No. 2 are more regular, there is still a wide variation. The question also arises whether the water-soluble potash so nearly represents the available potash in this class of materials as in fertilizers made up entirely of mineral ingredients. Since the fertilizer laws of some States recognize only the water-soluble potash, while the laws of other States permit total potash to be determined, this matter becomes of considerable importance.

With sample No. 3, the mixture of 9 grams of acid phosphate and 1 gram KCl gave an average of only 6.12 per cent, while the theoretical amount, based upon the analysis of the acid phosphate and the chemically pure KCl, was 6.35 per cent. The mixture of 9.5 grams of acid phosphate and 0.5 gram KCl gave only 3.07 per cent, while the theoretical was 3.23 per cent. The time at our disposal would not permit of an investigation into the cause of these low results.

While it was impossible to obtain as comprehensive results as were desired, owing to the late date when the samples were sent out, yet the results reported are sufficient to justify a continuation of the work along these lines in order to adapt the methods to these classes of fertilizing materials.

Mr. Huston stated that he was somewhat interested in the matter of the loss of potash and cited some work of the Kentucky Agricultural Experiment Station of several years ago, in which it was found that the more certain materials were boiled the more concentrated they became, and the less potash there appeared to be. He said for that reason the method of boiling described on page 21 of Bulletin 46 had been abandoned in his laboratory and the one substituted was as follows: Put 10 grams of the material in a nitrogen flask, graduated to 500 cc; add 200 cc of distilled water and force steam through the

fluid for 30 minutes. The steam is introduced by means of a small lead pipe reaching to the bottom of the flask. Mr. Huston stated that by the use of this method an increase in the volume of fluid was obtained as the solution went on and a higher percentage of potash secured.

Mr. SHUTT. In connection with the question of potash, it may be of interest to the members to know that we have found a very satisfactory method for the determination of potash in soils as follows: The potash being in solution (in hydrochloric acid in case of total potash or in 1 per cent citric acid solution in case of available potash), ammonia is added until the liquid is decidedly alkaline. Without any filtration, about two grams of chemically pure barium carbonate is now added and the whole boiled for about half an hour and filtered. To the filtrate, a few cc of ammonium oxalate, carbonate, and hydrate are added. After heating the substance is allowed to stand. The precipitated carbonates and oxalate are filtered off, the filtrate evaporated to dryness, and the residue gently ignited until ammonia fumes cease. If the ignited residue does not readily dissolve to a clear solution with a few drops of water, it is again treated with a few cc of hydrate and carbonate of ammonia, warmed and filtered, evaporated to dryness and gently ignited. The potash is now precipitated by platinic chlorid after acidulation of the solution by hydrochloric acid.

The report on nitrogen was next called for. Mr. Shiver stated that, owing to continued bad health, it was impossible for him to take an active part in the work on nitrogen and so he turned over the whole matter to Mr. Perkins, the associate referee. The president then introduced Mr. Perkins, who read the report on nitrogen as follows:

REPORT ON NITROGEN.

By W. R. PERKINS, *Associate Referee.*

I was notified by the president of this association, about the first of last March, that on account of ill health the referee, Mr. Shiver, would not be able to attend to the duties of the position. He requested me to take up the work for this season, and a similar request was made by Mr. Shiver about the same time.

Owing to the late date at which this order was received, and not knowing at what time the meeting would be held, the time at my disposal for becoming more familiar with the workings of the methods under consideration was very limited. Consequently, the instructions for work were in accord with Mr. Shiver's suggestions, being practically the same as for last year with regard to the determination of available organic nitrogen by the neutral permanganate and pepsin-hydrochloric acid methods.

Considerable difficulty was experienced in procuring the materials for use in the work, and I was unable to get samples of hoof and horn meal and leather in its various forms in any quantity, though a small amount of sole leather was obtained locally, and after much grating and grinding a sufficient quantity was prepared for three samples of the raw material, and a like number of a mixture containing leather. Samples of these were sent out to two chemists with the other samples.

Toward the latter part of March a letter was addressed to twenty-three chemists asking whether they desired to assist in the work or not. Favorable replies were received from eleven, and a set of samples was sent to each of them. The samples were as follows: No. 1, bat guano; No. 2, tankage; No. 3, ground bone; No. 4, fish; No. 5, dried blood; No. 6, cottonseed meal; No. 7, mixed fertilizer, containing 150 grams each of cotton-seed meal, fish, and dried blood, 200 grams of ground bone, and 925 grams of acid phosphate; No. 8, raw leather; No. 9, mixed fertilizer, containing 56 grams of raw leather, 50 grams each of dried blood and cotton-seed meal, and 300 grams of acid phosphate. All samples were passed through a millimeter sieve, thoroughly mixed, and the bottles filled from the same pile. The directions for work sent with the samples were as follows:

DIRECTIONS FOR THE A. O. A. C. WORK ON NITROGEN, 1900.

- (1) Thoroughly remix the material for analysis before beginning the work.
- (2) Determine total nitrogen by Kjeldahl method, using on No. 1 the method as modified for nitrates.

(3) Determine available nitrogen by neutral permanganate method (Street), as follows: Into a 600 cc Erlenmeyer flask weigh an amount of sample equivalent, approximately, to 0.075 gram of nitrogen. Digest this with 100 cc of permanganate of potash solution (16 grams of pure KMnO_4 to 1,000 cc of water) over a steam bath, or over water vigorously boiling, for thirty minutes. Shake thoroughly after ten minutes, and wash down sides of flask with a small quantity of same strength permanganate solution at same temperature of flask, using a fine-tipped wash bottle for this purpose. After this shake gently two or three times, and in case much of the substance adheres to side of flask, wash down with as little permanganate solution as possible. Remove from flask and add 150 cc of cold water, filter through a heavy folded filter, about 18 cm in diameter, and wash with cold water till total filtrate equals about 700 cc. Dry and determine nitrogen in residue by Kjeldahl method.

(4) If you have time, after completing the above, I would be pleased to have you determine available nitrogen by the pepsin-hydrochloric acid method, as follows: Weigh 1 gram of sample into a 150 cc flask and add 100 cc of pepsin-hydrochloric acid solution. This solution is prepared by mixing 5 grams of Parke & Davis's pulverized pepsin (guaranteed to dissolve 3,000 times its weight of freshly coagulated egg albumen) in 1,000 cc of hydrochloric acid diluted to a strength of 1 per cent.

The flasks are then kept for twenty-four hours loosely corked in a water bath having a constant temperature of 40°C . At the end of the second, fifth, eighth, and eleventh hours 2 cc of a 10 per cent hydrochloric acid solution are added, shaking the flask thoroughly at the same time.

After twenty-four hours digestion, filter, wash four or five times, using 20 to 30 cc of water for each washing, and determine nitrogen by the Kjeldahl method in the washed and dried residue.

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From the 11 laboratories to which samples were sent, partial or complete reports were received from 9, embracing the work of 11 chemists. The results of the work appear in the tables below:

TABLE I.—*Total nitrogen.*

Analyst.	Bat guano.	Tank- age.	Ground bone.	Fish.	Dried blood.	Cotton- seed meal.	Mixed fertil- izer No. 7.	Leath- er.	Mixed fertil- izer No. 9.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
F. B. Carpenter, Virginia....	11.64	7.64	3.65	8.38	13.44	7.28	3.30		
R. G. Newborn, Virginia	11.64	7.64	3.72	8.32	13.50	7.24	3.27		
J. A. Bizzell, North Carolina.	11.74	7.71	3.61	8.36	13.72	7.35	3.33		
	11.82	7.65	3.63	8.24	13.67	7.27	3.25		
J. W. Ames, Ohio.....	11.78	7.64	3.73	8.27	13.59	7.36	3.34		
T. C. Trescott, Washington, D. C.	11.59	7.62	3.71	8.25	13.24	7.15	3.35		
A. W. Ogden, Connecticut...	11.69	7.59	3.82	8.36	13.55	7.24	3.32		
	11.73	7.60	3.84	8.36	13.57	7.24	3.34		
O. W. Knight, Maine.....	11.49	7.76	3.71	8.40	13.49	7.36	3.41		
	11.59	7.70	3.77	8.34	13.57	7.36	3.41		
	11.65				13.45				
C. D. Holley, Maine	11.50	7.68	3.68	8.40	13.47	7.28	3.44		
	11.43	7.68	3.70	8.36	13.41	7.28	3.36		
	11.56	7.70	3.70	8.38	13.43	7.26	3.40		
		7.64	3.74	8.38	13.43		3.38		
E. B. Ferris, Mississippi	11.54	7.58	3.65	8.18	13.40	7.19	3.17	6.74	3.08
	11.54	7.52	3.65	8.18	13.42	7.18	3.19		
	11.52	7.62	3.68	8.24	13.46	7.12	3.25		
		7.53	3.70	8.24	13.36	7.11			
J. M. McCandless, Georgia ..	11.67	7.61	3.72	8.30	13.57	7.25	3.20	6.83	3.20
W. R. Perkins, Mississippi...	11.62	7.58	3.73	8.32	13.47	7.21	3.21	6.71	3.14
	11.70	7.58	3.67	8.34	13.40	7.25	3.19		
	11.70	7.55	3.66	8.26	13.46	7.15	3.21		
	11.58	7.49		8.27	13.36	7.21	3.24		
	11.57				13.67	7.21	3.24		
	11.59					7.15			
	11.60								
	11.55								
J. B. Robb, Maryland	11.56	7.44	3.82	8.24	13.40	7.22	3.37		
	11.58	7.52	3.77	8.30	13.34	7.18	3.38		
	11.68	7.50	3.83	8.30	13.48	7.24	3.45		
J. P. Street, New Jersey	11.51	7.44	3.82	8.13	13.26	7.18	3.20		
Average	11.61	7.60	3.72	8.30	13.46	7.23	3.30	6.76	3.14

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash.*

Analyst.	Bat guano.	Tank- age.	Ground bone.	Fish.	Dried blood.	Cotton- seed meal.	Mixed fertil- izer No. 7.	Leath- er.	Mixed fertil- izer No. 9.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. A. Bizzell, North Carolina.	2.96	0.91	1.32	0.97	0.86	0.70	1.26		
	2.92	.90	1.27	.91	.71	.60	1.24		
J. W. Ames, Ohio.....	2.66	1.03	1.31	1.02	.57	.99	1.63		
T. C. Trescot, Washington, D. C.	2.92	2.15	1.17	.96	1.06	.92	.88		
A. W. Ogden, Connecticut ..	2.48	.89	.93	.83	.52	.39	1.02		
	2.52	.93	.99	.86	.62	.52	1.15		
			1.05		.83	.60	1.17		
						.82	1.38		
O. W. Knight, Maine	2.71	.84	1.02	.87	.61	1.12	1.20		
	2.83	.80	1.04	.94	.54	1.00	1.17		
		.86				.96	1.19		
C. D. Holley, Maine	2.48	1.02	1.02	.86	.58	1.36	1.14		
	2.69	1.11	.99	.94	.60	1.38	1.18		
	2.81	1.15	.97	.94	.63	1.42	1.17		
		1.11				1.08			
		1.17				1.06			
E. B. Ferris, Mississippi	2.24	.73	1.06	.82	.67	.57	1.05	5.75	1.39
		.69	1.10	.87	.65	.52	1.07	5.80	1.45
						.47	1.14		1.42
							.96		1.44
W. R. Perkins, Mississippi...	2.30	.88	.96	.76	.67	.61	.94	5.85	1.09
	2.34	.80	.89	.81	.83	.75	.87	5.82	1.21
	2.40	.74	.76	.75	.87	.49	.83		
	2.56	.65	.81		.80	.49			
	2.55	.98	.82		.75	.84	.78		
	2.30				.75	.75	.94		
							1.03		
J. B. Robb, Maryland	2.64	.86	1.62	.75	.70	.72	2.00		
	2.50	.81	1.56	.84	.84	.70	1.96		
	2.63	.73	1.65	.81	.78	.59	1.92		
J. P. Street, New Jersey	2.73	.77	1.18	.65	.52	.45	1.33		
	2.66	.74	1.14	.74	.56	.45	1.25		

TABLE III.—*Availability of organic nitrogen by the neutral permanganate method.*

Analyst.	Bat guano.	Tank- age.	Ground bone.	Fish.	Dried blood.	Cotton- seed meal.	Mixed fertiliz- er No. 7.	Leath- er.	Mixed fertiliz- er No. 9.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. A. Bizzell, North Carolina.	74.87	88.15	63.53	88.31	93.72	90.42	63.33		
	75.21	88.28	64.91	89.02	94.86	91.79	62.42		
J. W. Ames, Ohio.....	77.41	86.51	61.87	87.66	95.80	86.41	¹ 51.19		
T. C. Trescot	74.80	¹ 71.80	68.46	88.36	¹ 91.99	87.12	¹ 73.43		
A. W. Ogden, Connecticut ..	78.82	88.29	75.71	90.07	96.16	¹ 94.61	69.36		
	78.48	87.76	74.15	89.47	95.42	92.80	65.46		
			72.58		93.88	91.71	64.86		
						88.53	58.55		
O. W. Knight	76.59	89.20	72.72	89.60	95.48	84.78	64.80		
	75.55	89.71	72.19	88.76	96.00	86.41	65.68		
		88.94				86.95	65.10		
C. D. Holley, Maine	78.43	86.70	72.43	89.71	95.68	¹ 81.31	66.47		
	76.61	85.52	73.24	88.75	95.53	¹ 81.04	65.29		
	75.56	85.00	73.78	88.75	95.30	¹ 80.49	65.58		
		85.52				85.16			
		84.74				85.43			
E. B. Ferris	80.57	90.34	71.13	90.01	95.00	92.02	67.18	14.56	54.87
		90.87	70.02	89.40	95.15	92.72	66.56	13.82	52.92
						93.42	61.25		53.89
						91.52			53.24
W. R. Perkins.....	80.20	88.34	74.05	90.83	95.02		¹ 70.71	12.55	61.34
	79.86	89.39	75.92	90.22	93.83	89.59	¹ 72.89	13.00	65.17
	79.34	90.06	¹ 79.45	90.95	93.54	93.19	¹ 74.14		
	77.96	91.24	¹ 79.61		94.06	93.19	¹ 73.52		
	78.05	87.02	¹ 78.10		95.38	88.33	¹ 75.69		
	80.20		¹ 77.81		94.43	90.41	67.91		
J. M. McCandless	79.70	89.22	74.83	90.72	94.32	90.90	66.89	13.76	¹ 49.27
	77.12	87.38	68.01	87.03	94.03	86.20	¹ 55.31	13.17	43.75
J. B. Robb	77.24	88.50	¹ 57.39	91.18	94.78	90.02	¹ 41.17		
	78.10	89.17	¹ 58.94	89.85	93.73	90.30	¹ 42.35		
	77.32	90.24	¹ 56.57	90.21	94.18	91.82	¹ 43.53		
J. P. Street	76.28	89.65	69.10	92.00	96.07	93.73	58.43		
	76.88	90.05	70.15	90.89	96.22	93.73	60.93		
Average	77.07	88.39	71.59	89.20	94.97	89.95	64.50	13.47	54.32

¹ Omitted from average.TABLE IV.—*Results on digestion of organic nitrogen with pepsin-hydrochloric acid.*

Analyst.	Bat guano.	Tank- age.	Ground bone.	Fish.	Dried blood.	Cotton- seed meal.	Mixed fertiliz- er No. 7.	Leath- er.	Mixed fertiliz- er No. 9.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. A. Bizzell.....	5.14	3.45	.28	2.64	.64	.53	.51		
		3.25	.29	2.80	.62	.58	.54		
T. C. Trescot	5.86	4.20	.25	3.20	.61	.55	.58		
A. W. Ogden	6.00	3.80	.19	2.78	.52	.48	.46		
	6.06	3.84	.20	2.78	.52	.48	.48		
W. R. Perkins.....	6.34	5.08	.36	3.76	.88	.79	.64	6.38	.92
	6.44	5.05	.32	4.13		.79			
		4.74		3.61					

TABLE V.—*Availability of organic nitrogen by pepsin-hydrochloric acid method.*

Analyst.	Bat guano.	Tank- age.	Ground bone.	Fish.	Dried blood.	Cotton- seed meal.	Mixed fertiliz- er No. 7.	Leath- er.	Mixed fertiliz- er No. 9.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
J. A. Bizzell.....	156.36	56.38	92.26	67.23	95.39	92.47	84.19		
T. C. Trescot.....	49.43	44.88	93.26	61.21	95.39	92.30	82.65		
A. W. Ogden.....	48.51	49.74	94.78	66.75	96.17	93.37	82.53		
J. M. McCandless.....	49.33		92.47	70.12	96.31	92.27	81.56	14.49	73.75
W. R. Perkins.....	45.44	132.71	90.27	152.50	43.47	89.00	80.12	4.92	70.70
	144.58	133.10	91.68	56.45		89.00			
						91.34			
Average.....	48.18	50.33	92.45	66.32	95.34	91.39	82.19	9.70	72.22

¹ Omitted from average.

NOTES BY THE ANALYSTS.

O. W. Knight, Orono, Me.—Found it impossible to remove residue completely from flasks after digestion with K_1MnO_4 solution, so 500 cc breakers were used instead for the majority of the samples.

C. D. Holley, Orono, Me.—Used 500 cc round-bottom flasks placed well down in steam bath and surrounded by live steam. Found it impossible to wash down sides of flasks thoroughly. Used 500 cc breakers with part of the work, stirring instead of shaking.

T. C. Trescot, Department of Agriculture.—With sample No. 6 the permanganate was entirely reduced before the expiration of 30 minutes.

J. P. Street, New Brunswick, N. J.—Made the digestion over a steam bath, and followed the instructions strictly in every respect. Thinks the addition of water to the flask before filtering a drawback on account of its retarding the determinations. Mr. Street also sends results of his work with thirty classes of organic materials by this method. Quite a number of determinations on bone, fish, tankage, blood, and leather are given and show very satisfactory results.

A. W. Ogden, New Haven, Conn.—Directions were followed closely on the permanganate, but on the pepsin-hydrochloric acid method the solution was made by dissolving 5 grams of Parke, Davis & Co.'s 1:3,000 pepsin in 1,000 cc of hydrochloric acid diluted to a strength of 0.2 per cent. The Connecticut station has always used this strength solution. Mr. Ogden gives a number of comparative analyses by the permanganate and pepsin-hydrochloric acid methods on bone, fish, tankage, blood, leather, cotton-seed meal, and castor pomace, which showed about the same relative figures as did the light materials in the official samples, except on bone. With this the average of two analyses was higher by the permanganate than by the pepsin-hydrochloric acid method. He also made duplicate determinations on five mixed fertilizers. These gave the availability of the nitrogen by the permanganate method from 25 per cent to 40 per cent higher than by the pepsin-hydrochloric acid method. The materials in both instances were washed with 300 cc of water before treatment with the solution.

DISCUSSION OF RESULTS.

The directions for work on total nitrogen were followed, so far as reported, by all of the analysts taking part in the work except Mr. Trescot, who used the Gunning method. There is considerable difference between the highest and lowest results with every sample in Table I, being between 0.2 and 0.3 per cent on all except the

bat guano and dried blood and 0.39 per cent and 0.48 per cent respectively for them. With the care taken in mixing the materials these variations are difficult to account for from this cause alone, though we recognize the fact that a lack of uniformity in samples may always be looked for as a possible source of error in comparing work of this kind. It is noticeable from the tables that there is a tendency throughout in the results of some of the analysts to be either high or low on all of the samples.

Rejecting a few of the highest and lowest results in Table II, the bat guano, bone, fish, dried blood, the two mixed fertilizers, and the leather, appear to agree quite closely, while with the tankage and cotton-seed meal the figures are more variable. The analysts, however, with few exceptions, got very concordant results on their different determinations, indicating that under the same conditions the method is very reliable.

In the digestion of the samples as followed in this work, the cotton seed meal completely destroys the color of the permanganate before the expiration of the thirty minutes and the tankage approaches very near to that result. Under these conditions it is very evident that any variation in the quantity of solution used will be attended by some change in the results. A lack of accuracy in measuring the prescribed quantity or differences in the amount of solution used in washing down sides of flasks might account for some of the wide variations in this table. The suggestion as to washing down materials adhering to flasks was made after doing some work on a sample of bone meal, with which changing the quantity of permanganate solution had very little effect on the quantity of nitrogen undissolved, even though as much as 200 cc were used.

The figures in Table III are derived by computation from those of Tables I and II. They show the determined availability as calculated from the average of each analyst's results on total nitrogen, and the separate determinations of that element left undissolved. The results, unlike those in the preceding table, show the greatest variations with the bone and two mixed fertilizers, all low nitrogen. The results with bat guano, tankage, fish, dried blood, cotton-seed meal, and leather, omitting five results, show to much better advantage. In expressing these results in "per cent of nitrogen available," the differences are very much magnified with samples of low nitrogen content, and results on such materials must agree within very narrow limits to be of any value. A difference of 0.2 per cent of undissolved nitrogen in blood would mean about 1.5 per cent in its availability. Such a difference in results on bone, or the mixed fertilizers above, would mean fully 6 per cent when calculated to per cent of nitrogen available, and in samples containing 2 per cent of nitrogen the difference would be 10 per cent.

The mixed sample No. 7 was made of the same raw material as that distributed for this work. Using the averages on these materials given in Tables I and II, its computed analysis should be:

	Per cent.
Total nitrogen.....	3.23
Availability of nitrogen.....	87.27

As determined by the different analysts it was:

	Per cent.
Total nitrogen.....	3.30
Availability of nitrogen.....	64.50

Mixed fertilizer No. 9, in like manner contained, as computed:

	Per cent.
Total nitrogen.....	3.10
Availability of nitrogen.....	72.00

As determined:

	Per cent.
Total nitrogen.....	3.14
Availability of nitrogen.....	54.32

The difference in the determined availability of these two samples as compared with their computed availability is very great, and was undoubtedly due to the presence of soluble phosphoric acid.

To test the effect of the presence of this substance on the results of some of these samples by this method, two sets of determinations were made. The same quantity of material as used in the regular work was weighed out, and from 2 to 3 grams of acid phosphate added to each, otherwise they were treated as in the official work. The amount of nitrogen in residues after digesting and washing was:

Tankage.	Bone.	Fish.	Blood.	Cotton-seed meal.
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
2.67	1.91	4.71	10.50	1.41
2.80	1.98		10.23	1.86

It was noticed in treating the raw materials that the filtrate from the residues after digestion with permanganate solution gave a precipitate with a water solution of soluble phosphate. This probably explains the high results just given, as well as the unsatisfactory results with the mixed fertilizers of the tables.

As bearing on this point, the following results would indicate the necessity of washing the samples of mixed fertilizers before treatment with permanganate solution:

TABLE VI.—*Results on organic nitrogen in mixed fertilizers by the neutral permanganate method after washing sample.*

Analyst.	Material.	Amount of material used.	Availability.
		<i>Grams.</i>	<i>Per cent.</i>
J. P. Street	Mixed fertilizers	2.34	78.50
J. P. Street	do	2.34	77.84
A. W. Ogden	do	2.00	82.00
W. R. Perkins	do	2.30	87.27

While these results do not agree as closely as could be desired, they approach much nearer to the theoretical figures than do those of this sample in Table III.

A glance at the few results in Tables IV and V will show that with the pepsin-hydrochloric-acid method the results on bone, dried blood, cotton-seed meal, and the mixed fertilizers are very close, but with the bat guano, tankage, and fish they do not agree at all, even after omitting the very low results of my own work on these samples.

In conclusion, it must be said that the work for this year has not resulted in any material advance toward solving the problem under consideration, and I very seriously doubt if either the neutral permanganate or pepsin-hydrochloric acid method can be so perfected as to render them of much value in detecting adulterations in the nitrogen supply of fertilizers. Twenty-six per cent of the nitrogen in sample No. 9, for instance, is supplied in raw leather, so great an amount that the presence of the material is unmistakable, yet the availability of the nitrogen in the mixture, as determined in the raw material by the permanganate and pepsin-hydrochloric acid methods, is 72 per cent by the former and 70.6 per cent by the latter, considerably higher than bat guano, tankage, or fish by the pepsin method and higher than bone by the permanganate method. With less leather, as would un-

doubtedly be the practice of fertilizer manufacturers using it, the results would be still higher, and, consequently, there would be more confusion.

I believe, however, that the permanganate method, to which my work has been largely confined, can be made to give good results in the hands of different analysts by carefully conducting the work with reference to the following points:

- (1) Weigh out the same amount of material and wash thoroughly in case of mixed fertilizers.
- (2) Use the same quantity of solution, not washing down sides of flasks.
- (3) Keep a uniform temperature.
- (4) Bring all of the material under the action of the solution for the full time.
- (5) Avoid the passing of the finer particles of the substance through the filter in washing.

I would suggest that the succeeding referee give the above points his consideration in any further work on this method, and recommend that the neutral permanganate method (Street) and the alkaline permanganate method (Jones) be referred to the referee for 1901.

The president then introduced Mr. Dyer, of London, England, who addressed the association as follows:

ADDRESS OF MR. DYER, OF LONDON, ENGLAND.

GENTLEMEN OF THE ASSOCIATION: I thank you for this kindly greeting. It is a great pleasure to me and a privilege to meet you all here, because I have followed closely and with much interest during the last half dozen years the work you have been doing on this side of the water, in connection, more especially, with soil work. For a considerable length of time I have tried to make some advancement in our methods of determining the available constituents in soils. I published a paper on the subject, and that paper at once met with interest on every hand on this side of the Atlantic. You at once set to work here to use my methods and to try them comparatively with other methods on parallel lines. My own work, which has been on availability, has been almost entirely confined to mineral constituents. This question of available nitrogen, which is occupying the attention of the meeting at the present moment, is one to whose discussion I am afraid I can not offer any useful contribution, but as your president has been so kind as to call upon me, I can not let this opportunity pass of expressing my great pleasure, the great delight it has been to me to see you taking up my little bit of work as you have here and doing so much which I hope will in the end prove to be the means of improving methods and making an advance upon the steps which I have endeavored to make.

I am in America at the present time as the representative of the Rothamsted Agricultural Experiment Station, with which I am not officially connected, but where I have done a great deal of work, and I am here to deliver the Lawes lectures. I may say that in the Rothamsted laboratory considerable interest has been taken by Sir Henry

Gilbert in the papers of Professor Hilgard and his associates on the determination of available nitrogen in soils in the work which has been done by many of you on the lines sketched out by Hilgard. Large numbers of Rothamsted soils have been subjected in the laboratory to investigation by Hilgard's method, or rather by the association's modification of what is generally known as the Hilgard method. I am afraid I can not say that the results in the investigation of the soils which we have at Rothamsted promise to give us very much light. We find that the proportion of nitrogen soluble in dilute alkali seems to bear a pretty constant relation to the total nitrogen in the soil, and the relationship between the nitrogen and the soluble organic matter does not appear to throw any definite light upon the comparative availability of the nitrogen. We do not get very much more information than would be given from the total nitrogen, but that is, of course, comparing a number of different soils, originally of the same type, that have been modified by the process of cropping and manuring.

I thought you would like to know that as you take an interest in methods which we sometimes originate on our side, so we at the same time follow your work and interest ourselves in the methods you put forward. I thought you would like to know that the association's method for determining nitrogen in soils had been subjected to examination by us.

The PRESIDENT. The association is very glad to hear from Mr. Dyer and we are glad to have him join in the discussion of our reports and papers.

Mr. Withers then presented the following paper:

THE RATE OF NITRIFICATION OF CERTAIN FERTILIZERS.

By W. A. WITHERS and G. S. FRAPS.

The value of any fertilizer depends on its availability to the plant, that is, the readiness with which it can be taken up directly by the plant or converted into forms which can be assimilated. Nitrogen can be assimilated directly by ordinary agricultural plants in the form of certain organic compounds, ammonium salts, and nitrates, nitrates being the preferable form. Different plants behave differently toward these forms; some are not benefited by ammonium salts when young, and others are; some feed upon nitrates during their entire period of growth, and others are not benefited by them until they have attained some size. Nitrates, however, appear generally to be most valuable.

When combined nitrogen, in whatever form of combination, is placed in the soil, it is converted into ammonium salts, nitrites, and finally nitrates, with greater or less speed, depending on the form of combination, the temperature, condition of the soil, etc., provided certain living organisms are present, and they generally are. If in any given soil we determine the relative rate with which nitrogenous fertilizers, which can not be utilized directly by the plant, are converted into nitrates, it should throw some light upon the relative values of these particular fertilizers to plants.

Müntz and Girard (Central-Blatt f. agr. Chem.: 20, 656 (1891) abs.) have determined the rate of nitrification of certain fertilizers. A small quantity of the fertilizer was intimately mixed with natural soil, and kept at the temperature of 15°–25° C., properly moistened, and at the end of a given period leached with water, and the nitrate determined in the extract. The nitrate existing in the soil at the beginning of the experiment was previously determined. The time was thirty, thirty-two, and thirty-nine days for different sets. The nitrogen converted into nitrates and the nitrogen recovered from the soil by horse-tooth corn in two years is shown in the following tables:

Percentage of nitrogen oxidized and amount thereof recovered by the crop of two successive years.

Fertilizer.	Nitrified in 30 days.	Recovered by corn.
	<i>Per cent.</i>	<i>Per cent.</i>
Ammonium sulphate	75.0	76.7
Dried blood	72.4	55.0
Roasted horn, fine.....	71.0	60.1
Flesh meal.....	70.4	
Horn trimmings, fine	55.5	53.3
Poudrette, rather coarse	18.1	18.1
Roasted leather, fine.....	11.6	38.3
Leather chips, raw.....	0.39	

In another series the order of nitrification was as follows, the substance nitrified to the greatest extent being given first: Bat guano, dried grasshopper, dried cockchafers, flesh meal, dried blood. There is very little difference between the three substances named last. The nature of the soil had a great influence on the change. Nitrification was most active in a light soil from Jourville, used in the experiment referred to above, next in a garden soil, then in a chalky soil, then in a marled moor soil. Very little nitrification occurred in a very calcareous clay except with cow manure and yellow lupines, which loosened its texture, and none in an acid moor soil, with the same two exceptions.

P. Bonâme (Exp. Sta. Record: 9, 732, abs.) determined the nitrates in the drainage water from a sandy soil deficient in lime to which fertilizers had been added. The order of nitrification at the end of the first month was found to be—fish guano (most rapid), blood, fertilizer, oil cake, and ammonium sulphate. When calcium carbonate was added nitrification took place more rapidly, but the order was still dried blood, oil cake, ammonium sulphate.

In the experiments performed by us the fertilizing materials were those sent out by the referee of the association for this year to test the methods for determining the availability of nitrogen. A quantity of material equivalent to 0.6 gram of nitrogen was intimately mixed with 1,000 grams of soil, the sample being sifted through a coarse sieve, 6 meshes to the inch. The soil was then placed in precipitating jars and kept in a dark closet, adding enough water to make the percentage 11.6. At suitable periods three of the jars were weighed, and the loss of water replaced. The temperature was 28°–30° C., and the time was 3 weeks. When calcium carbonate was added the amount was just sufficient to combine with the nitrogen of the fertilizer if the entire amount were converted to nitric acid. At the end of the experiment the nitrates were leached out, and the amount determined by the Tiemann-Schulze method. The amount of nitrates in a blank experiment was deducted from the total.

On account of the small percentage of ammonium sulphate nitrified in the first

series, the experiments with cotton-seed meal and ammonium sulphate were repeated, the time being twenty-six days, the temperature 23°–26° C., and the sample being moistened as before. The soil was taken from the same field as in the first series, but differed from it somewhat, as is shown by the fact that it contained 0.1641 gram of nitrogen per kilogram as nitric acid, whereas the former sample contained only 0.0595 gram.

As to the availability of nitrogen, we have selected the results obtained by vegetation tests with oats and Hungarian grass by Jenkins and Britton (Connecticut State Station Report, 1897, p. 257) and in the laboratory of the North Carolina station with the same materials nitrified by Bizzell, using the pepsin-hydrochloric acid and the neutral permanganate methods. These results are shown in the following table:

Comparison of the results obtained by vegetation tests, pepsin hydrochloric acid and neutral permanganate methods in determining the availability of nitrogen.

Fertilizer.	Rate of nitrification (time, 3 weeks).				Availability.		
	Without CaCO ₃ .		With CaCO ₃ .		Soluble in KMnO ₄ .	Soluble Pepsin-HCl.	Vegetation tests.
	Per cent.	Rank.	Per cent.	Rank.			
					<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Dried blood	34.8	100	54.9	100	94.4	94.7	73.3
Cotton-seed meal.....	33.9	97	54.8	100	91.1	91.1	64.8
Second series.....	26.7	-----	-----	-----			
Dried fish.....	30.3	87	46.5	85	88.7	67.3	63.9
Tankage	26.2	75	34.8	63	88.3	56.4	49.4
Bat guano	22.4	64	35.8	65	75.1	56.4	-----
Bone	18.9	54	16.6	30	64.2	92.3	16.7
Ammonium sulphate....	1.3	4	31.1	55	100.0	100.0	-----
Second series.....	3.4	-----	32.6	-----			
Nitrate of soda	-----	-----	-----	-----	100.0	100.0	100.0

It will be seen by an examination of the table that there is a very close correspondence between the order representing the rate of nitrification during three weeks and that representing the availability as shown by chemical and vegetation tests, except in the case of sulphate of ammonia. It seems to be nitrified much more slowly than the organic compounds used, and yet in many experiments in the field it gives results closely approximating those given by nitrate of soda. These results give rise to some interesting questions in regard to the availability of nitrogen in its different forms and also in regard to nitrification. A more detailed account of this work will be found in Bulletin No. 176 of the North Carolina station. This investigation will be continued by us.

MR. FREAR. What was the character of the soil with which you mixed these materials, as to basicity or acidity?

MR. WITHERS. I do not think we made this test. It was a soil which had been under cultivation for some time, was not very rich, and not much organic matter in it.

MR. FREAR. Was it rich in basic material?

MR. WITHERS. No; it did not have much lime, and when we added lime to the soil, nitrification took place much more rapidly than in other circumstances. There was not an excess of lime in the soil.

Mr. EWELL. I would like to ask if you offer any reason for the low rate of nitrification of ammonia in this case?

Mr. WITHERS. I suppose I can not offer any explanation that would be conclusive, but one explanation that seems to be indicated by it is this: It is reasonably supposed that organic compounds change first their ammonia salt and then change the nitrate. It would seem that the ammonia salt would undergo nitrification much more quickly than any form of organic compound. It may be that the difficult decomposition of the ammonium sulphate in the soil would account for the fact that the nitrification of the ammonia in ammonium sulphate would not take place as rapidly as nitrification of organic compounds.

Mr. WITHERS then cited the figures in another experiment along the same lines.

Mr. EWELL. What would be the concentration of the ammonia in the soil?

Mr. WITHERS. Six-tenths gram of nitrogen to 1,000 grams of soil. We had the same quantity of nitrogen in every case.

Mr. FREAR. In this connection, if it is the ammonia which produces this effect, why would not the effect be intensified with an increased quantity of calcium carbonate? The effect might have been due to sulphuric acid which was set free. Of course acid will accumulate in proportion to the quantity of nitrogen which has been acted upon.

Mr. EWELL. I do not think there is any doubt about the acid.

Mr. FREAR. The effect of ammonium sulphate fertilizers has been so thoroughly demonstrated that it need scarcely be mentioned. Our own experiments have been continued about twenty years on the same soil. We have found the nitrate ammonium sulphate plots to be very distinctly acid. Clover is crowded out and even the grass is pretty well crowded out, and the ordinary red sorrel has come to take its place. On examining these plots for nitrogen, I find we have a large excess of nitrogen. This may be due to the soil, but it may also be due to the failure of the ammonia to be nitrified.

Mr. Wheeler here remarked that the results obtained by Mr. Withers were so remarkable he would like to ask what kind of pots the experiments were conducted in.

After some further discussion on the subject the following paper was presented by Mr. Williams.

VARIABLE AMMONIA RESULTS IN MIXED FERTILIZERS.

By C. B. WILLIAMS.

As the determination of ammonia in commercial fertilizers is one of great importance on account of the high valuation of ammonia, it was thought that it would not be amiss to present at this meeting of the association some results obtained during the spring of 1899 in the laboratory of the fertilizer control of the North Carolina Experiment Station.

It has been, and still is, the custom of the control to prepare all fertilizers by grinding until they will pass through a sieve with perforations 1.25 millimeters (20 mesh) in diameter and then thoroughly mix with a spatula. Frequently it was found that on samples prepared in this way it was absolutely impossible to obtain concordant results, no matter how carefully the work was performed. It was not uncommon to find that duplicates weighed out at the same time and worked in exactly the same way gave results as divergent as 1 per cent. At first it was thought that these abnormal differences were due to leakages in the nitrogen distillation apparatus, but upon thorough inspection it was seen that this could not be the cause, as all connections were tight and secure. After many futile efforts to detect the cause of these great differences, it was finally observed that in every case the variations occurred on high grade complete fertilizers, whose ammonia was derived from either blood, fish scrap, tankage, or nitrate of soda.

Now, when such fertilizers are prepared by being forced through apertures 1.25 millimeters in diameter and then mixed, the relative coarseness of the materials furnishing nitrogen is so great as compared with the other components of the fertilizer as to make it absolutely impossible to obtain a homogeneous sample for analysis, even when the greatest care is exercised in mixing.

Further, it was found that the heterogeneity of the samples constantly increased after preparation, due to the tendency of these materials to separate from the others when the nitrogenized material used was either blood, fish scrap, or tankage. On account of their coarse mechanical condition and light specific gravity upon agitation, a constant concomitant of weighing, they will rise to the top of the sample. With these conditions, it is very easy to see that the ammonia results will vary directly with the quantity of this coarse nitrogenized material, weighed out for each determination. When a fertilizer bottle containing one of these fertilizers is tilted the coarse, light particles on top of the sample will roll to the lower side and the results will depend upon whether you take the portion for analysis from the upper or lower side of the tilted bottle. When this was noticed a portion of the coarse lower part and a portion of the fine upper part from almost all the samples were selected by the spatula and weighed separately and analyzed. By this selection it was not infrequent to obtain results differing by as much as 1 or 2 per cent of ammonia. In the subjoined table the results in the third column are from the fertilizers when they were prepared by being passed through a sieve with perforations 1.25 millimeters (No. 20) in diameter, while those in the second are samples that have passed a sieve with perforations 0.625 millimeter (No. 40) in diameter.

Fertilizer No.	Preparation.		Fertilizer No.	Preparation.	
	Passed through sieve with perforations 0.625 mm. in diameter.	Passed through sieve with perforations 1.25 mm. in diameter.		Passed through sieve with perforations 0.625 mm. in diameter.	Passed through sieve with perforations 1.25 mm. in diameter.
	<i>Per cent NH₃.</i>	<i>Per cent NH₃.</i>		<i>Per cent NH₃.</i>	<i>Per cent NH₃.</i>
1077.....	5.15	5.68	1280.....	2.33	1.94
1077.....	5.20	4.44	1280.....	2.21	² 2.25
1077.....	5.22	4.76	1280.....	2.19	¹ 2.40
1077.....		4.88	1280.....		¹ 2.96
1077.....		4.76	1297.....	4.52	4.76
1077.....		4.93	1297.....	4.42	4.65
1077.....		4.78	1297.....		4.20
1077.....		¹ 5.40	1297.....		4.83
1077.....		² 4.55	1303.....	2.65	2.62
1094.....	7.50	7.08	1303.....	2.70	2.78
1094.....	7.48	6.96	1303.....		2.43
1094.....	7.53	6.77	1303.....		2.50
1094.....	7.58	6.81	1192.....	2.99	2.59
1094.....		7.20	1192.....	3.01	² 2.53
1094.....		7.89	1192.....	3.01	¹ 2.80
1111.....	5.66	5.60	1222.....	3.88	3.56
1111.....	5.61	5.79	1222.....	3.91	¹ 4.59
1111.....	5.71	5.63	1222.....	3.96	² 3.40
1111.....		5.37	1222.....	4.02	3.71
1111.....		² 5.55	1227.....	2.74	2.55
1111.....		¹ 6.01	1227.....	2.77	2.63
1111.....		¹ 6.90	1227.....		² 2.55
1183.....	8.86	8.32	1227.....		² 2.70
1183.....	8.81	² 8.72	1227.....		¹ 3.28
1183.....		¹ 9.14	1236.....	3.08	3.06
1183.....		¹ 9.34	1236.....	3.18	3.16
1183.....		8.18	1236.....	3.20	² 2.77
1190.....	3.04	2.43	1236.....		¹ 3.75
1190.....	3.04	2.63	1254.....	5.15	4.88
1190.....	3.13	2.77	1254.....	5.15	5.17
1190.....	3.06	² 2.32	1254.....	5.22	² 5.00
1190.....		¹ 2.91	1254.....		¹ 6.07
1366.....	8.69	9.12	1324.....	2.77	2.50
1366.....	8.69	8.94	1324.....	2.74	¹ 3.05
1366.....	8.64	8.75	1324.....	2.70	
1366.....		8.89	1076.....		6.73
1366.....		8.84	1076.....		6.51
1366.....		8.85	1076.....		6.90
1366.....		8.91	1076.....		6.22
1182.....	8.01	7.66	1076.....		6.80
1182.....	7.98	² 6.37	1076.....		6.29
1182.....		¹ 8.33	1076.....		6.58
1182.....		² 7.65	1081.....		8.72
1182.....		¹ 8.15	1081.....		8.33
1258.....	2.84	2.66	1081.....		8.72
1258.....	2.77	² 2.96	1081.....		8.81
1258.....		¹ 3.08	1180.....		6.07
1258.....		2.96	1180.....		² 6.17
1277.....	3.08	2.74	1180.....		¹ 6.85
1277.....	3.06	² 3.30	1180.....		6.39
1277.....	3.11	¹ 4.10	1180.....		¹ 7.17
1277.....	3.06				

¹ Selected with spatula, the coarse part for analysis. ² Selected with spatula, the fine part for analysis.

Upon a study of the table it is seen that where the samples were forced through a No. 40 sieve the lowest difference was 0.02 per cent ammonia, and the highest was 0.14 per cent; while the results obtained when the samples were passed through a No. 20 sieve gave as the lowest difference 0.27 per cent, and the highest 1.96 per cent. In all these determinations the modified Kjeldahl method was used with 0.7 gram substance.

Even upon a superficial glance at the results in the table it is easy to see the necessity of at least reducing our fertilizers to a fine mechanical condition before attempting to make an analysis of them. There is not the slightest doubt in my mind that to reduce fertilizers containing fish scrap, blood, tankage, or nitrate of soda to a homogeneous state they must be forced through a sieve with apertures not much larger than those of the No. 40 sieve. It seems fair to assume that not only are the ammonia results affected by the mechanical condition of fertilizers as in the above, but also those of potash and phosphoric acid. It is hoped that during the present fertilizer season this work may be supplemented by that on more samples.

Mr. MYERS. As some of you know, I have an interest in the nitrate of soda to the extent of promoting its use as much as possible, and I encounter this difficulty, which you gentlemen can correct if you will, and that is the manufacturers of commercial fertilizers complain that the State chemists fail to give them credit for nitrate of soda when mixed with other goods. The nitrate of soda, or any nitrate for that matter, is certainly the most available form of nitrogen, and I think the day is not far distant when the State chemists of this country and the chemists of other countries will find it to the interest of their inspection to state the nitrogen composition of a fertilizer just as you do the phosphoric-acid composition of a phosphate. Some of the stations are already doing it in this country, and I wish I could induce you to consider the advisability of presenting this composition of your fertilizers more clearly in your reports. I do not want to criticise in any way or ask for unnecessary favors, or anything of that kind, but this is based on scientific grounds, and where a manufacturer is using an available form of nitrogen, as in the case of a nitrate, he certainly should have credit for it.

I want to say further that there will be much more nitrate, probably, in this spring's fertilizers than in those of last spring, for the reason that, owing to the proficiency in handling the raw materials, nitrate is now probably the cheapest ammoniated material in the market, and it is very desirable that manufacturers should have credit for using it.

Mr. WITHERS. Mr. Wheeler has said, "These results are so at variance with those which are usually obtained." I would like to know to what results he has reference.

Mr. WHEELER. I intended to say that the results are so at variance with those we consider as the usual experience. I, perhaps, did not use those words, but that is what I intended to state.

I am about to indorse what the last speaker has said in regard to the nitrate of soda. The ground taken is strictly correct upon a scien-

tific basis. In some States there may be pressure to complete the work in as short a space of time as possible, and, therefore, there are inducements to leave out determinations which should be made, not only in the interests of those who are selling good goods, but in the interest of the consumer. I think the association should express a sentiment in regard to this matter. I move that it is desirable that the form of nitrogen should be determined and reported on in the analysis of fertilizers.

Motion seconded and carried.

Mr. HUSTON. Two years ago a committee of experiment station chemists presented a report on uniform methods of standard fertilizer analysis. All forms of nitrogen were included in that report. Now, before we can arrive at any very satisfactory state of affairs in regard to this, we must do some work at home, in the various States. There is no State law existing for making a distinction between the forms of nitrogen. In my own State it is simply total nitrogen.

Mr. FREAR. In Pennsylvania we make only total nitrogen. There are so many fertilizers sold under different brands, I fear fewer analyses of the brands would be made than the farmers would want if we attempted to make three determinations of nitrogen instead of one.

Mr. Shutt then referred to some work done in his laboratory on soils containing different percentages of water and gave a description of the method in use for the determination of nitric and nitrite nitrogen in soils as follows:

One hundred grams of the fresh undried soil is placed in a wide-mouth bottle and 1,000 cc of ammonia-free water added. The mixture is kept well shaken for one hour; it is then allowed to stand one hour. Two hundred and fifty cubic centimeters are now decanted and distilled and the distillates nesslerized in the usual manner and recorded as free ammonia. Another 250 cc is at the same time decanted and placed in contact with a zinc copper couple. After 24 hours this is distilled and the distillates nesslerized. From the results the amount of ammonia obtained the day previously is subtracted and the difference calculated to nitrogen present as nitrates and nitrites.

Mr. McDONNELL. From the remarks of Mr. Myers, I do not quite understand what the trouble is in the case of the manufacturer. Is it that the chemist is not able to get the entire amount of nitrate of soda? In our experience there might be two sources of error; one the difficulty of getting the nitrate out of the sample by the Gunning or Kjeldahl method, as modified for nitrates, and another is that there is certainly sometimes a loss of nitrogen from the loss of nitrate in fertilizers due to the presence of acid phosphate. In regard to the other matters, nitrate of soda being cheaper, there would be no trouble about using plenty of nitrate.

Mr. MYERS. I suppose this calls for a little explanation. The claim of the manufacturers is that, in the first place, the methods of analy-

sis do not distinguish between the forms of nitrogen and indicate the nitric nitrogen. In the next place it is admitted on all hands that if you mix nitrate of soda with a strictly acid phosphate, of course there will be a loss. That is a technical difficulty that can not be overcome, and I suppose will not be for some time. But the point we wish to make is, that we desire, as soon as it can be done without discommoding the State chemists any more than possible, we desire, I say, to get the chemists to state what brands of fertilizer have nitrate in them. It is not every year that nitrate is cheaper than other sources of nitrogen. This year it is due to the trust in ammoniating materials.

MR. MACFARLANE. I would like to say in regard to this discussion that the fertilizer act in Canada obliges the manufacturer, when he sends in a standard sample every year for examination and analysis, to state the materials that have been used in its manufacture. This is published along with the results of analysis. We never make any statement as to what fertilizers should contain nitrates, but in a case where nitrates have been found, by the test which this gentleman has spoken of, it is mentioned.

MR. FREAR. This matter has received some consideration at my hands, but I have feared this result: The test is such that the introduction of a very small quantity of nitrate would cause all the goods to be classed as nitrates, as soon as the manufacturers found what was being done.

MR. BARTLETT. All that our State law requires is the total nitrogen. Of course we are not obliged to make the other determinations, but for a few years we made both the determination of nitric nitrogen and ammonia, and found we could not afford to do it. In adopting this plan we took 1 gram of the fertilizer and leached on the filter paper, in the same manner as for determining soluble phosphoric acid, and then determined the nitrogen in the rest. In this way we obtained the soluble nitrogen and that determination, in combination with the test for nitric nitrogen, would be valuable. The soluble nitrogen is to be stated instead of nitrogen as nitrate or ammonia.

MR. VAN SLYKE. For several years we have been publishing water soluble nitrogen. That includes, of course, something more than nitrates, but it has satisfied the farmers and we have heard no criticism from the manufacturer.

MR. WINTON. We have with us on this occasion a representative of the nitrate industry. A few years ago a gentleman attended the meeting who represented the ammonium sulphate industry. In our State we have always determined total nitrogen and nitrogen in the form of ammonium salts. Would it not be well to do that in an official way? In other words, would it not be well to reconsider that motion which was proposed to include ammonium salts? Not that I believe ammo-

nium salts are equal in value to nitrate of soda, but it simply comes in the composition of a fertilizer.

MR. DAVIDSON. I think we should determine the availability. There are a great many States that do not have a valuation for the different forms of nitrogen. There is no doubt that this is a serious defect in the laws of those States. The States ought to adopt a uniform system of stating the content of nitrogen of the various fertilizers that are put upon the market. I think we should have a law requiring, first, that all three forms should be determined, and, second, that when organic matter is stated availability also should be given. Let us add to that motion that the different forms of nitrogen be valued according to their availability, and that nitrate of soda, cotton-seed meal, dried blood, etc., be given their true valuation.

MR. HUSTON. We have a blank for application for fertilizer analysis, on the original manufacturer's sample, and in order to relieve us a little our work was definitely stated to be on fertilizers containing nitrate. I find a large number make affidavit that the fertilizers contain nitrate when there is not a trace of nitrate in them. They have a habit of making a claim for anything which is suggested to them.

MR. KILGORE. I would like to mention one matter, which was suggested in my report this morning, in regard to a uniform statement of fertilizer analyses and the branding of bags, etc. We had this matter up before the Association of Commissioners of Agriculture of the Southern States and they adopted part of the report, the same one that this Association has also adopted, in regard to the statement of analysis. Further, it is their opinion, on the recommendation of the chemists, that the manufacturers should make a claim of the materials out of which a fertilizer is made. A great many manufacturers objected to this, and in fact the commissioners were very doubtful as to whether such a demand should be made upon them. I believe the fertilizer committee of this Association might take up this matter, as well as that of the determination of the forms of nitrogen, and report on both subjects.

MR. SHIVER. In order to dispose of this matter I would like to make a motion. I would like to say, in regard to the referee's report, that on account of neglect on my part we failed to have any work done on the Jones method for determining the availability of nitrogen. Those who were at the California meeting will remember that I recommended that the Jones method and the Street method be further tried by the association. I would say that I have never in my limited experience obtained very satisfactory results from the Jones method, especially with cotton-seed meal. This may be due, however, to lack of experience on my part. I make the motion that the Jones and Street methods be recommended for further trial next year.

REPORT ON PHOSPHORIC ACID.

By E. G. RUNYAN, *Referee*.

In response to a letter inviting cooperation in the work on the phosphoric acid samples for the current year, twenty-two chemists expressed a willingness to take up the work. To each was sent a set of three samples, with a copy of the following instructions:

INSTRUCTIONS FOR PHOSPHORIC ACID WORK FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR 1900.

Three samples have been prepared for the work on phosphoric acid for 1900, as follows:

No. 1 is ground phosphate rock.

No. 2 is American slag.

No. 3 is ground phosphate rock.

DETERMINE TOTAL PHOSPHORIC ACID IN ALL THE SAMPLES by the following methods:

(1) *Official gravimetric method* (Bull. 46, Div. of Chem., U. S. Dept. of Agri., p. 12).

(2) *Optional volumetric method* (Bull. 46, Div. of Chem., U. S. Dept. of Agri., p. 13), or the following modification:

Make solution of the phosphate according to one of the official methods (Bul. No. 46, Div. Chem., Dept. of Agric., p. 12). Take an aliquot portion containing not more than 30 mg. of P_2O_5 , add NH_4OH in excess, then HNO_3 in slight excess, then an excess of the official molybdate solution. Place in a water bath at 45° to 50° C. for one-half hour with occasional stirring, filter, and wash with water until 25 cc of the filtrate fails to neutralize one drop of the standard potassium hydroxid solution. Transfer filter and contents to the beaker, add about 30 cc of water and sufficient standard alkali to dissolve the yellow precipitate, titrating the excess of alkali with standard acid, adding 1 cc of a 1 per cent solution of phenolphthalein as indicator. See Bulletin No. 46, Division of Chemistry, Department of Agriculture (p. 13), for directions for preparing the standard acid and alkali solutions.

(3) *Volumetric method*, precipitating by shaking at room temperature.

Proceed as in the last preceding method to the point where the solution is ready to be placed in the water bath. Then cool solution to room temperature, add molybdate solution at the rate of 75 cc for each decigram of P_2O_5 present, place flask containing solution in shaking apparatus and shake for thirty minutes at room temperature, filter at once, wash and titrate as in preceding method.

DETERMINE IRON AND ALUMINUM in the three samples, by the following methods:

(1) *Modified acetate method*.—Dissolve two grams of sample in 30 cc HCl and 10 cc HNO_3 (all iron must be in the ferric condition), cool, make up to 250 cc, filter. Take 50 cc, dilute to 400 cc, nearly neutralize with NH_4HO , using a few drops of mythyl orange solution as indicator, heat to 70° C., and add 25 cc of a 25 per cent solution of ammonium acetate. Filter and wash with a hot 5 per cent solution of ammonium nitrate. Ignite and weigh the iron aluminum phosphate. Fuse the ignited precipitate with sodium carbonate, dissolve in dilute HNO_3 and determine the phosphoric acid in the usual way. Determine the iron in 50 cc of the original solution by titrating with permanganate (Margerite's method, Fresenius Quant. Anal., p. 267, ed. 1881).

(2) DETERMINE ALUMINA by precipitating with phenylhydrazine, as follows: Make solution of the phosphate in the usual way. Take an aliquot portion, dilute to 250 cc, heat to near boiling, and add ammonia slowly, as long as the precipitate formed redissolves readily. A neutral saturated solution of ammonium bisulphite is then added, drop by drop, with stirring, to the hot solution, until it becomes colorless, showing the complete reduction of the iron. Then add phenylhydrazine, a little at a time, until the precipitation is complete. The precipitate of aluminum hydroxid and aluminum phosphate is collected in a filter, washed with warm water containing a small amount of phenylhydrazine bisulphite, until the wash water is free from chlorids or from iron, as indicated by $(NH_4)_2S$. Dry the filter and precipitate, ignite, heat to bright red, cool and weigh in covered crucible, as the precipitate is very hygroscopic. Fuse the mass with sodium carbonate, dissolve in hot dilute nitric acid and determine the P_2O_5 in the usual way.

The weight of the total precipitate less the weight of P_2O_5 found gives the weight of alumina. The iron is determined in another portion of the original solution by titrating with potassium permanganate.

To prepare the saturated solution of ammonium bisulphite to any given volume of strong ammonia, add an equal volume of water and pass sulphur dioxide into the cooled solution until the solution becomes yellow.

The wash solution is prepared as follows: To a few cubic centimeters of phenylhydrazine in a beaker a saturated water solution of sulphur dioxide is added gradually until the precipitate of phenylhydrazine sulphite, which at first separates out in crystals, is redissolved to a yellow solution. If after a few minutes an odor of sulphur dioxide is perceptible, a few drops of phenylhydrazine are added to neutralize this excess of sulphurous acid. Use 10 cc of this concentrated solution to 100 cc of water for washing the alumina precipitate.

When the method employed differs from directions given, please note the fact. Results on other samples will be gladly received and full credit given in the report.

Please report results at least one month before the date of the association meeting.

Results on the determination of total phosphoric acid in the samples sent out have been received from 19 analysts, representing 11 laboratories. These results are given in Table I. Where more than one result by the same method was reported by an analyst, the individual results are given in column "a" and the average of such results in column "b." When but one result was reported it is given in column "b."

TABLE 1.—*Per cent total P_2O_5 found in referee's samples.*

Analyst.	Sample No. 1.				Sample No. 2.				Sample No. 3.			
	Official gravi- metric method.		Optional volu- metric method.		Official gravi- metric method.		Optional volu- metric method.		Official gravi- metric method.		Optional volu- metric method.	
	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)
J. W. Ames, Ohio Agricul- tural Experiment Sta- tion.....	14.33											
	13.67	14.51	13.65	14.30	18.75	17.41	17.30	17.62	27.02	26.35	26.55	27.19
	13.70	13.69	13.70		17.44	17.12	17.12		26.29	26.65	26.65	
	13.70	13.68	13.60		17.61	17.35	17.35		26.43	26.55	26.55	
A. W. Blair, Florida Agri- cultural College.....			13.75		17.46	17.27	17.27		26.34	26.60	26.60	
						17.30	17.30					
						17.27	17.27					
Charles P. Beistle, Penn- sylvania Agricultural Experiment Station ..	13.50	13.51	13.62	13.55	16.94	16.87	16.87	16.86	26.40	26.50	26.50	26.45
	13.52	13.57	13.52	13.32	16.92	16.85	16.85		26.52	26.45	26.45	26.60
				13.20	16.97							26.40
M. L. Bloomberg, Rich- mond, Va.....		13.46						16.86				
F. B. Carpenter, Rich- mond, Va.....								16.84				
E. M. Chace, U. S. Depart- ment of Agriculture..	13.95	13.51	13.60	13.63	17.15	17.40	17.40	16.86	26.10	26.46	26.50	26.16
			13.65	13.70		17.50	17.50				26.55	26.55
			13.95	13.50		17.45	17.45				26.60	26.60
E. B. Ferris, Mississippi Agricultural and Me- chanical College.....	13.71	13.60	13.60	13.45	17.39	17.30	17.30	17.20	26.46	26.50	26.50	26.30
	13.66	13.69	13.65	13.45	17.53	17.28	17.28	17.20	26.43	26.50	26.50	26.30
	13.71	13.66	13.63	13.50	17.53	17.35	17.35	17.25	26.51	26.50	26.50	26.30
			13.70	13.50	17.42	17.40	17.40	17.25				26.30
			13.68			17.50	17.50					26.30
			13.72									

¹ Precipitated by maintaining in bath at 45° to 50° for thirty minutes.

TABLE I.—*Per cent total P₂O₅ found in referee's samples—Continued.*

Analyst.	Sample No. 1.				Sample No. 2.				Sample No. 3.			
	Official gravi- metric method.		Optional volu- metric method.		Official gravi- metric method.		Optional volu- metric method.		Official gravi- metric method.		Optional volu- metric method.	
	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)	(a)	(b)
C. Glaser, Baltimore, Md.	13.78	13.78	13.59	13.26	414.52	414.09	513.99	26.12	26.82	26.12	26.82	25.90
O. W. Knight, Maine Ag- ricultural Experiment Station	14.23 14.21	14.22	14.21	14.16	17.99 18.04 18.14	17.82 17.82 17.82	18.03	27.08 27.16	27.19	27.04 26.99	27.04 26.99	27.02
F. C. Lamb, Raleigh, N. C.	13.47 13.46 13.39 13.38	13.43	17.22	17.19 17.33	17.25	25.96 25.98 25.91	25.96	25.95	25.96	25.95	25.96	25.96
J. R. Laughlin, Mary- land Agricultural Col- lege	13.56 13.67 13.69	13.64	13.40 13.48 13.50	17.31 17.31 17.25	17.29	17.05 17.10 17.12	17.05	25.80 25.93 25.97	25.80 25.90 25.80	25.80 25.90 25.80	25.80 25.90 25.80	25.80
C. C. McDowell, Clemen- son Agricultural Col- lege, South Carolina	13.75 13.84 13.89 14.05 14.00 13.98	13.86	13.85 13.80 13.85 13.75 13.80 13.80	13.75 13.65 13.70	17.62 17.44 17.38 17.42	17.41	17.35 17.40 17.35 17.30 17.35 17.30	26.52 26.62 26.31 26.52 26.38 26.82	26.70 26.70 26.85 26.80 26.80 26.75	26.80 26.65 26.65 26.45	26.80 26.65 26.65 26.45	26.01
H. K. Miller, Florida Ag- ricultural College	14.05 14.05 14.10 13.72 13.80 13.90 13.85 13.90 13.90	13.87	14.05 14.05 14.10 13.72 13.80 13.90 13.85 13.90 13.90	13.87 13.87 13.87 13.87 13.87 13.87 13.87 13.87 13.87	17.40 17.40 17.60 17.50 17.35 17.30 17.55 17.35 17.35	17.41	17.45 17.40 17.60 17.50 17.35 17.30 17.55 17.35 17.35	26.60 26.60 26.60 26.60 26.55 26.70 26.55 26.60 26.65	26.60 26.60 26.60 26.60 26.55 26.70 26.55 26.60 26.65	26.60 26.60 26.60 26.60 26.55 26.70 26.55 26.60 26.65	26.60 26.60 26.60 26.60 26.55 26.70 26.55 26.60 26.65	26.01

E. L. Parker, Richmond, Va.....	13.60	13.60	13.68	17.23	17.46	16.82	17.18	17.30	26.03	26.15	26.15	26.04
B. F. Robertson, Clemson Agricultural College, South Carolina.....	13.90	13.60	13.70	17.23	17.46	17.18	17.40	17.30	26.70	26.19	26.70	26.80
		13.60	13.90	17.29	17.26	17.30	17.30	17.40	26.22	26.60	26.60	26.75
		13.60	13.80	17.29	17.26	17.50	17.35	17.35	26.09	26.60	26.75	26.60
E. G. Runyan, U. S. Department of Agriculture.....	13.61	13.61	13.80	17.23	17.40	17.42	17.40	17.30	26.20	26.75	26.60	26.50
	13.63	13.50	13.50	17.30	17.30	17.40	17.40	17.40	26.55	26.50	26.50	26.50
	13.53	13.50	13.60	17.55	17.50	17.55	17.55	17.40	26.55	26.55	26.55	26.50
	13.53	13.60	13.70	17.55	17.50	17.55	17.55	17.40	26.55	26.55	26.55	26.50
M. N. Straughn, Maryland Agricultural College.....	13.48	13.45	13.70	17.27	17.25	17.18	17.25	17.10	25.93	25.80	25.80	25.85
	13.51	13.55	13.53	17.32	17.32	17.18	17.32	17.10	26.05	25.99	25.95	25.85
	13.64	13.60	13.60	17.37	17.37	17.37	17.37	17.37	26.51	26.47	26.43	26.25
	13.80	13.73	13.50	17.56	17.56	17.35	17.35	17.23	26.51	26.50	26.50	26.25
	13.74	13.73	13.52	17.59	17.55	17.35	17.35	17.23	26.43	26.50	26.50	26.25
W. S. Welch, Mississippi Agricultural and Mechanical College.....	13.75	13.75	13.51	17.49	17.55	17.33	17.33	17.23	26.55	26.53	26.53	26.25
	13.70	13.70	13.51	17.55	17.55	17.33	17.33	17.23	26.55	26.53	26.53	26.25
	13.75	13.75	13.51	17.56	17.56	17.33	17.33	17.23	26.55	26.53	26.53	26.25
	13.73	13.73	13.51	17.56	17.56	17.33	17.33	17.23	26.55	26.53	26.53	26.25
	13.55	13.45	13.45	17.21	17.21	17.35	17.35	17.35	26.01	26.50	26.50	26.25
	13.43	13.50	13.50	17.23	17.23	16.88	16.88	16.93	26.10	26.50	26.50	26.25
C. B. Williams, Raleigh, N. C.....	13.51	13.49	13.49	17.27	17.26	16.90	16.92	16.90	26.11	26.06	26.06	25.86
	13.51	13.51	13.51	17.27	17.26	16.90	16.92	16.90	26.11	26.06	26.06	25.86
	13.51	13.51	13.51	17.27	17.26	16.90	16.92	16.90	26.11	26.06	26.06	25.86
	13.51	13.51	13.51	17.27	17.26	16.90	16.92	16.90	26.11	26.06	26.06	25.86
Averages.....	13.75	13.70	13.63	17.33	17.40	17.24	17.26	17.26	26.31	26.44	26.44	26.41
Number arranged.....	18	17	12	12	17	16	11	11	18	17	17	12

¹ Precipitated by maintaining in bath at 45° to 50° for thirty minutes. ² Precipitated by maintaining in bath at 65° for thirty minutes. ³ Contaminated with iron.

⁴ Not included in averages. ⁵ Precipitated by maintaining in bath at 45° to 50° for thirty minutes; not included in averages.

⁶ Washed with water at 50°; not included in averages.

COMMENTS BY ANALYSTS.

C. P. Beistle.—The two volumetric methods were slower than the official gravimetric method, and also gave less uniform results. A great deal of time was required in washing the yellow precipitate with 10 per cent nitric and 3 per cent ammonium nitrate, and water successively. If the precipitate was washed with water alone the results were invariably high, indicating the presence of molybdic acid in the precipitate. In precipitating the phosphoric acid at room temperature with shaking, some of the precipitate adhered to the sides of the flask, and could not be removed.

C. Glaser.—Concerning the titration of the yellow precipitate, I wish to say that the method is very good, but I can not rely upon getting always the same results. I do not succeed in washing out completely with cold water, but tepid water (50°) does very well.

J. R. Laughlin.—In the modified volumetric method I had trouble with the yellow precipitate running through filter. This was remedied by precipitating at higher temperature, or leaving in the bath for a longer time than given in the referee's directions. I precipitated for 30 minutes at 65°.

C. B. Williams.—The solution of the three samples of the referee's phosphoric acid was effected by first boiling 2 grams each in 200 cc flasks with 30 cc of hydrochloric acid (1.20 specific gravity) to a concentration of 10 or 15 cc, and then adding, after slightly cooling, 30 cc nitric acid (1.42 specific gravity), and again boiling until the cessation of the evolution of red fumes, which required about 10 or 20 minutes. This is the method used in this laboratory for effecting the solution of phosphoric acid in slags and fertilizers containing iron and alumina.

The volumetric results were obtained by precipitating in the cold in Erlenmeyer flasks with molybdic solution, and then placing the flasks in a revolving shaking machine for 30 minutes, 45 to 55 revolutions per minute being maintained. This method of precipitating gives a coarse precipitate that can be filtered and washed easily and quickly. It will be observed that the gravimetric results on sample No. 2 are perceptibly higher than those volumetrically determined. This is probably due to the presence of iron in the magnesium pyrophosphate, as a qualitative test for iron revealed its presence in considerable quantities in the "white precipitate."

OBSERVATIONS BY REFEREE.

By a comparison of the data reported it was found that all results by Mr. Glaser on No. 2, the sample of slag, were about 3 per cent lower than those reported by other analysts. A correspondence with Mr. Glaser brought out the fact that the solution of this sample in aqua regia was evaporated to dryness, and the residue taken up with hot dilute hydrochloric acid to separate the silica. This subsequent treatment with hydrochloric acid ought to have resulted in a solution of all the phosphoric acid, but it appears that in this case it failed to do so. These results are given in the table with this explanation, and will serve to emphasize a fact that has been observed before in the work of this association; that is, a slight variation in the method may cause a wide difference in the result.

By a reference to the table it will be seen that the results reported by two of the analysts are uniformly high in comparison with the other results received. With these exceptions the percentages by the several methods agree fairly well, and may be considered quite satisfactory. The gravimetric results in the sample of slag are slightly higher than those by the volumetric method, due probably to the presence of iron in the white precipitate. For this reason it is quite probable that the volumetric results on such material more nearly express the true percentage of phosphoric acid present.

One-half the analysts reporting on the optional volumetric method state that the precipitation was made in a bath at 45° to 50° for 30 minutes, as indicated in the table by a reference to footnote "1". This is the method of precipitation usually employed in the laboratory of the Division of Chemistry, as we have found that the yellow precipitate formed under these conditions filters rapidly and there is less danger of its contamination with molybdic acid.

Two years ago Mr. Kilgore, then referee on phosphoric acid, presented to this association some results obtained by precipitating the phosphoric acid at room temperature by shaking. One advantage claimed for this method was that there was less liability of the precipitate being contaminated with molybdic acid. The results reported at that time were so promising for this method that I requested a trial of it by the chemists taking part in the work this year. An inspection of Table I shows that while the results by shaking in the cold are very slightly lower, still they agree well with results by the other two methods. One analyst reports that in precipitating by shaking in the cold, a portion of the yellow precipitate adhered to the flask and could not be removed. This objection can be overcome by dissolving the adhering precipitate in a portion of the standard alkali, and then rinsing the contents of the flask into the beaker in which the titration is made, or by transferring filter and contents to the flask and making the titration in the flask.

Some trials were made by your referee to determine the time required to precipitate all the phosphoric acid by shaking in the cold. On several different days determinations were made in quadruplicate, one set of four shaken for 30 minutes and the other set shaken for 1 hour. All results agreed closely. The average results by the two periods of shaking differed by less than 0.05 of 1 per cent, showing that all the phosphoric acid was precipitated at the end of 30 minutes. Judging from my own experience and from the data reported by others, I believe this method capable of yielding good results in the hands of any careful analyst.

Some results, obtained by modifications of the official method, were reported by Mr. Welch on the referee's samples, on an acid phosphate practically free from iron, and on chemically pure sodium phosphate. These figures are given in Table II.

TABLE II.—*Results on phosphoric acid by modifications of official methods. By W. S. Welch.*

Methods.	Referee's samples.			Acid phosphate.	C.p. sodium phosphate containing 19.83 per cent P_2O_5 .
	No. 1.	No. 2.	No. 3.		
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Official gravimetric.....	13.75	¹ 17.75	26.47	17.88	
				17.91	
Official gravimetric after precipitating the "yellow" precipitate by shaking 30 minutes at room temperature.....				17.86	
				17.91	
Official gravimetric, adding 5 cc ammonium citrate solution before adding magnesia mixture.....	13.56	17.36	26.28	17.76	
	13.64	17.44		17.72	
Official gravimetric, adding 10 cc ammonium citrate solution before adding magnesia mixture.....				17.73	
				17.71	
Official gravimetric, adding 2 grams citric acid to 0.25 gram sample before adding magnesia mixture.....		17.32			
		17.33			
	13.51	17.20	26.25	17.70	19.85
				17.80	
Volumetric, shaking 30 minutes at room temperature.....				17.75	
				17.83	
				17.80	
				17.80	
Optional volumetric.....	13.73	17.34	26.53		
Official gravimetric, "white" precipitate dissolved in HCl, reprecipitated with NH_4OH				17.64	
				17.67	

¹ Contaminated with iron.

IRON AND ALUMINA IN PHOSPHATES.

Results on iron and alumina in the phosphoric-acid samples sent out were received from seven analysts representing as many laboratories. These results are brought together in Table III.

TABLE III. — *Results on iron and alumina in referee's samples.*

Analysts.	Sample No. 1.				Sample No. 1.				Sample No. 3.			
	Per cent Fe_2O_3 by perman- ganate.		Per cent Al_2O_3 .		Per cent Fe_2O_3 by perman- ganate.		Per cent Al_2O_3 .		Per cent Fe_2O_3 by perman- ganate.		Per cent Al_2O_3 .	
	(a)	(b)	Acetate method.	Phenylhydra- zine.	(a)	(b)	Acetate method.	Phenylhydra- zine.	(a)	(b)	Acetate method.	Phenylhydra- zine.
J. W. Ames, Ohio Agri- cultural Experiment Station.....	4.52	4.78	8.22	7.00								
C. P. Boiste, Pennsyl- vania Agricultural Ex- periment Station.....	4.45	4.49	8.27	8.25								
A. W. Blair, Florida Agri- cultural College.....		4.78	7.50	7.44	17.83	2.41	56.71	1.60	4.24		5.16	
			7.38	7.48	1.31	1.07			7.93		8.10	
E. B. Ferris, Mississippi Agricultural and Me- chanical College.....	3.37	3.40	8.35	18.34					8.00		7.30	
	3.43		8.32						5.22			
			8.35						4.00		5.08	
C. Glaser, Baltimore, Md.		6.28	6.52	8.45	21.22				4.00		15.01	
	4.33		7.86	8.98	18.23	1.84			4.81		4.97	
E. G. Runyan, United States Department of Agriculture.....	4.33	4.34	8.07	8.74	18.21	1.92	1.91	1.58	5.09		8.44	
	4.43		8.34	8.85		1.97		1.75	4.99		8.24	
								1.75	5.04		8.61	
S. H. Shell, Richmond, Va.....		5.00	27.10		18.12	21.22			5.75		26.05	
			36.47			30.06			34.55		44.16	
			46.90			40.01						
Average.....		4.72	7.46	8.59	18.69		1.24		5.11		7.50	
Difference between high- est and average.....		1.56	.79	.15	2.53		.67		.87		.94	
Difference between low- est and average.....		1.32	.94	.14	.86		1.03		1.11		.53	
Number averaged.....		7	5	2	5		3		6		4	
												2

¹ Precipitated twice, with addition of a little sodium phosphate the second time; not included in averages.² Contained lime; not included in averages.³ Precipitated twice; not included in averages.⁴ By acetate method as used in laboratory of Virginia-Carolina Chemical Co.; not included in averages.⁵ Not included in averages.

F. B. Carpenter (S. H. Sheib).—The iron and alumina determinations were made by the modified acetate method, as described in the referee's letter of instructions, and also by the modified acetate method used in this laboratory, which is as follows:

Weigh two grams of sample in a 200 cc flask, add 35 cc strong hydrochloric acid and boil thirty minutes. Dilute, cool, make to mark, mix, filter through dry filter. Take 50 cc for analysis, add a few drops nitric acid, boil, add ammonia to alkalinity and a few drops of methyl orange, then acidify slightly with hydrochloric acid. Heat to 70° and add 15 to 20 cc of a strong hot solution of ammonium acetate made distinctly acid with acetic acid. Filter at once on folded filter, wash three or four times with a 5 per cent solution of ammonium nitrate and once with hot water. Dissolve the precipitate with dilute warm hydrochloric acid and reprecipitate the iron and alumina as above. Filter, wash six times with 5 per cent ammonium nitrate solution, dry, ignite, and weigh as iron and aluminum phosphate. When but one precipitation was made in the work on the referee's samples, the iron and aluminum phosphates contained heavy traces of lime, while two precipitations reduced the lime present to a slight trace.

C. Glaser.—The determination of the combined iron and aluminum oxids in sample No. 2, by precipitation of the iron and aluminum phosphate with acetate, proved an utter failure on account of lack of phosphoric acid. The phenylhydrazine process is excellent, but I can not account for the difference in results obtained, No. 1 being too high and No. 3 being too low.

OBSERVATIONS BY REFEREE.

The results on iron and alumina are rather discordant, although they show slightly closer agreement than did the results obtained the last time this subject was before this association.

The phenylhydrazine method is one proposed by Hess and Campbell, and your referee requested a trial of it in the hope that it might yield concordant results in the hands of the members of this association. As only one analyst outside of the Department of Agriculture reported results by this method, it would be manifestly unwise to base any conclusion on such meager data.

The other method given was the well-known acetate method with a slight modification. Two fertile sources of error by the acetate method, as usually employed, are the solvent action of the wash water on the precipitate of iron and aluminum phosphate, and the contamination of the precipitate with lime. The modification proposed sought to reduce the solvent action to a minimum by washing with a 5 per cent solution of ammonium nitrate and to avoid the error due to the presence of lime by taking not more than 0.4 gm of the sample and diluting to 400 cc before precipitation.

In some preliminary trials of this method in the laboratory of the Division of Chemistry, on solutions of chemically pure salts, it was observed that no lime was precipitated by ammonium acetate in a faintly acid solution when the dilution was carried to the point mentioned above.

Some results by the two proposed methods on a solution of chemically pure aluminum sulphate and a mixture of chemically pure salts containing known quantities of alumina, iron, lime, and phosphoric acid are given in Table IV.

TABLE IV.—Results on alumina in mixtures of chemically pure salts, by E. G. Runyan.

Methods.	Mixture chemically pure salts containing Fe_2O_3 , P_2O_5 , CaO , Al_2O_3 .	Chemically pure aluminum sulphate.
	Per cent Al_2O_3 .	Per cent Al_2O_3 .
Theory.....	4.85	19.38
Phenylhydrazine method	$\left\{ \begin{array}{l} 5.14 \\ 5.18 \\ 5.18 \end{array} \right\} 5.17$	$\left\{ \begin{array}{l} 18.84 \\ 19.04 \\ 19.60 \end{array} \right\} 19.16$
Modified acetate method	$\left\{ \begin{array}{l} 4.70 \\ 4.60 \\ 4.72 \\ 4.65 \end{array} \right\} 4.67$	$\left\{ \begin{array}{l} 19.63 \\ 19.25 \\ 19.63 \end{array} \right\} 19.50$

The results on the chemically pure aluminum sulphate do not agree as closely as one could wish, while the average of the results on the mixture of pure salts by the phenylhydrazine method are 0.32 of 1 per cent too high, and by the acetate method 0.18 of 1 per cent too low.

Although the results reported on iron by the permanganate method do not agree as well as they should in the hands of experienced analysts, still I believe this method is the best one for the determination of iron in phosphates. As the alumina by the acetate method is determined by difference, it follows that an error in the determination of iron or phosphoric acid would cause a corresponding error in the percentage of alumina found. Some of the apparent discrepancies in the percentages of alumina as reported by the acetate method may be due to errors in the determination of iron. While the results given do not warrant the drawing of any definite conclusions, still I believe that some form of the acetate method will prove to be the best method for alumina.

The subject of the determination of iron and alumina in phosphates is one that, in my opinion, may well receive some attention from the members of this association during the coming year.

Mr. WHEELER. I should like to present a paper for the purpose of calling the attention of the association to some work which has been done on phosphoric acid.

Mr. Wheeler presented his paper as follows:

INCREASED ACCURACY IN PHOSPHORIC ACID DETERMINATION.

By H. J. WHEELER.

Berzelius¹ recognized the existence of an ammonium magnesium phosphate of the ortho type, richer in ammonia than the normal one. Subsequently Neubauer,² apparently unaware of Berzelius's observation, directed attention to such a salt, and, in recognition of its existence and the consequent error liable to arise in the determination of phosphoric acid, proposed the use of certain factors to correct the errors in analysis.

Gooch and Austin³ have shown that if the ammonium magnesium phosphate is

¹ Jahresb., 3 Jahrgang (1824) Uebersetzt von C. G. Gmelin, p. 92. Cited from Gooch and Austin.

² Zeit. f. Angw. Chemie (1896) pp. 435-440.

³ Am. Jour. of Science 7, pp. 187-198.

precipitated in the absence of an excess of ammonium hydroxid and ammonium salts and under definite conditions the normal salt is obtained. Their procedure obviates the necessity of applying factors for correcting results. It does away, also, with the necessity of adding magnesia mixture drop by drop in an attempt to bring about a compensation of errors.

There seem to be three excellent reasons why this association should give special consideration to the work of Gooch and Austin and why certain modifications of the present official method of determining phosphoric acid are advisable.

(1) By a modification of the method in accordance with their procedure the laboratory may be freed from the strong odor of ammonia.

(2) The laboratory expenses may be materially reduced by lessening the amounts of ammonium chlorid and ammonium hydroxid required.

(3) By the proposed modification close approximations to theoretical results are obtained, since the final precipitation is always conducted under essentially identical conditions.

As determinations are now conducted by the official method the large quantities of ammonium hydroxid and of ammonium salts, together with their varying relation to the magnesium salt present, render it impossible either to obtain the ideal salt or to secure a condition under which the deficiency of magnesia in the residue is compensated for by the magnesium oxid thrown down. According to the method of the association, one is directed to dissolve the yellow precipitate "on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc." Hydrochloric acid is then added until the solution is nearly neutral, before precipitating with magnesia mixture. Under such conditions large amounts of ammonium salts are not only present in all cases, but the quantity is necessarily variable. The existing conditions are further aggravated by the large quantities of ammonium hydroxid and ammonium chlorid in the official magnesia mixture.¹ After the precipitation with magnesia mixture is completed, the method of the association calls for the addition of 30 cc of ammonium hydroxid of 0.96 specific gravity. Since the strength of the ammoniacal solution and the quantity of ammonium salts at the time of precipitation is seldom twice alike, it must be obvious that if a nearly correct result is obtained finally, it must be due to chance and to a balancing of errors.

In addition to the points which have been enumerated, Gooch and Austin have shown that the double magnesium and ammonium phosphates are extremely insoluble, even in a faintly ammoniacal solution, and that such a solution may be used for washing the precipitate in the place of the disagreeably strong one provided for in the official method. The following is a modification of the official method whereby a precipitate of ideal composition may be obtained:

After dissolving the yellow precipitate in the usual manner the solution is allowed to cool (with care the precipitation may be made immediately), and the phosphoric acid precipitated with magnesia mixture made as directed by Blair. If it seems advantageous, the solution may be nearly neutralized with hydrochloric acid before precipitating. The magnesia mixture may be added at once instead of drop by drop. As soon as the precipitate subsides, the supernatant liquid is decanted upon the same asbestos filter upon which the final precipitate is to be collected. Most of the liquid may thus be poured off without the transference of more than a minimum portion of the first precipitate to the filter. The precipitate is dissolved in the least possible amount of hydrochloric acid with the addition of 75 cc to 100 cc of distilled water. Ammonium hydroxid is added until the solution reacts distinctly alkaline. The precipitate, even where but small quantities of phosphoric acid are present, is formed immediately. It subsides quickly and can be filtered in a few moments, or as soon

¹Two liters contain 280 grams of ammonium chlorid and 700 cc of ammonium hydroxid (0.96 specific gravity). Blair's mixture, on the other hand, contains in the same volume but 58 grams of ammonium chlorid and only sufficient ammonium hydroxid to give it a distinct odor of ammonia.

as the solution is cool. The wash water should be rendered faintly alkaline with ammonium hydroxid.

The following are a few data obtained by Miss Austin at the Rhode Island station in the analysis of ordinary commercial fertilizers by the official method and by the modification just described:

Comparison of methods in determining phosphoric acid.

Fertilizer No.		Official method.	Modified method (Blair's mixture).	Modified method: Precipitation from the warm ammoniacal solution (Blair's mixture).
		$Mg_2P_2O_7$ grams.	$Mg_2P_2O_7$ grams.	$Mg_2P_2O_7$ grams.
284	Solution I	.0742	.0720	
		.0728	.0712	
	Solution II	.0740	.0718	
		.0724	.0720	
286	Solution I	.0718	.0734	.0730
		.0714	.0738	.0732
	Solution II	.0720	.0734	.0732
		.0716	.0734	.0734
288	Solution I	.0788	.0788	.0788
		.0794	.0790	.0786
	Solution II	.0785	.0766	.0770
		.0786	.0762	.0772
318	Solution I	.0146	.0148	
			.0146	
	Solution II	.0144	.0146	
			.0146	

It will be seen from the foregoing that in some instances the results by the modified method were higher and in others lower than by the official method, but a better average agreement between parallel tests was obtained by the modified than by the official method.

When one considers that by the proposed modifications the magnesia mixture may be added at once instead of drop by drop, also that a great saving in ammonium chlorid, ammonium hydroxid, and hydrochloric acid may be effected, and that the disagreeably strong ammoniacal wash water may be replaced by a very weak one, there are abundant reasons, in addition to the attainment of greater accuracy, why the question of changing the official method is worthy of the serious consideration of this association.

MR. MACFARLANE. An apology is due this association for introducing the vexed question of available phosphoric acid. My paper is on that perplexing subject. I may say that we have in Canada, for the past twelve years at least, been loyal to Bulletin 46, and in fact every one of our analysts throughout the Dominion is furnished with a copy of the work in question, and its use is recommended and in fact urged. We have had no occasion during the period mentioned to suggest any changes, but it has happened that our peace paradise has now been dis-

turbed. We have had in Canada very large importations of basic slag, or Thomas phosphate, which I understand is very seldom imported into the United States, and the consequence is we have gotten into trouble. We are obliged to do something to give satisfaction both to consumers and manufacturers, not only of basic slag, but of other acidulated phosphates that may be sold in the Dominion.

Under our law we collect samples and they are submitted to analysis. Our district analysts applied the processes described in your methods of analysis to the basic slags, the consequence being that the amount of available phosphoric acid, which the law requires in phosphates sold in Canada, was perhaps underestimated. It did not come up to the 8 per cent of available phosphoric acid which, by law, the fertilizers in Canada should contain. Instead of having the supposed 8 per cent, it sometimes did not amount to more than 4 or 5 per cent, and the consequence was that the district analysts were obliged to characterize the basic slags which were being sold in the Dominion as adulterated, according to the act. Of course, this was not at all agreeable to the importers or venders of the basic slag, and we have heard a great deal of discussion, which culminated this last session of Parliament, in an appeal, as it were, to the agricultural committee of the House of Congress. What actually happened before that agricultural committee is detailed in our last bulletin, of which I have some copies here, and which I think a good many members of the association received directly. I shall therefore not refer again to what took place before that committee, but I will try to describe the action which I took subsequently. In order that I may not omit any of the important points, allow me to read from this memorandum:

MEMORANDUM FOR THE MEETING OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT WASHINGTON, D. C., ON THE 16TH OF NOVEMBER, 1900.

On pages 56 and 57 of Bulletin No. 70, issued by the Laboratory of the Inland Revenue Department at Ottawa, there will be found a statement regarding the present position of the question which has arisen in Canada as to the proper method of estimating the "available" phosphoric acid in basic slag, or Thomas phosphate powder. The importers of this article, represented by James Domville, esq., M. P. for Kings County, New Brunswick, did not accept the amendment to the fertilizers act proposed by the minister of inland revenue and approved by the agricultural committee of the House of Commons, and consequently the public analysts of Canada will find themselves face to face next year with the same conditions which now obtain, and be obliged to characterize nearly all samples of basic slag as "adulterated under the act."

Foreseeing the inconveniences of such a result, I took advantage of my stay in London, England, during the months of June and July, this year, to make an application to the Society of Public Analysts there for information as to whether it would be likely to take any action in the direction of establishing a uniform method for the determination of the available phosphoric acid in fertilizers. This application was

considered at a meeting of the council of the society, and the following gentlemen appointed a committee to consider the subject: Dr. Bernard Dyer, Dr. Voelcker, Messrs. John Hughes, and Alfred Sinesham. Owing to the lateness of the season, it was found impossible to arrange for a meeting of this committee in July, and the matter had therefore to stand until after the recess, with what result since I am unable to say.

While in London I thought it wise also to call on the firm of H. & E. Albert, chemical works, one of the largest shippers of basic slag to Canada, in order thoroughly to explain the position of our branch and why their wish could not be complied with, namely, that the Wagner method of determining the available phosphoric acid should be adopted in our laboratory. My arguments were mainly founded on the position taken under paragraph 5 on page 6 of Bulletin No. 70, which is as follows:

To apply a 2 per cent citric-acid solution in the manner described by Wagner to basic slag only and not to the water-insoluble part of other fertilizers would, without doubt, occasion strong objections on the part of the fertilizer manufacturers of Canada and of the United States.

I did not content myself with defending this position, but also suggested a method by which all fertilizers could be tested in exactly the same way without doing any injustice to basic slag or Thomas phosphate powder. This consisted in boiling the water-insoluble residue with a strong solution of ammonium chlorid, so as to remove any free lime which the sample might contain, and to do this before determining the available phosphoric acid, either by neutral citrate of ammonia or by a 1 per cent solution of citric acid, as recommended by Dr. Bernard Dyer and others. The gentlemen to whom I spoke in the London office of Albert's works proposed that I should discuss the method suggested with the chemists of the firm at Biebrich on the Rhine and with Professor Wagner in Darmstadt. This I ultimately consented to do, and the London house arranged for appointments with the parties named.

My interview with Dr. Wagner took place on July 12, and resulted most satisfactorily. We discussed the fertilizer act of the Dominion, the whole circumstances connected with the analysis of basic slag, and also my suggestion for treating it with a solution of ammonium chlorid. The latter he declared to be new and well worth a minute investigation, which he thought should be undertaken after consultation with the Albert firm. The authorities of the latter I also visited at their works at Biebrich, including not only Mr. Heinrich Albert, but the manager also, and the chief chemist, Mr. König. Mr. Albert made many objections to my proposal, more especially maintaining that the ammonium chlorid solution would dissolve out phosphoric acid. Experiments by Mr. König showed this fear to be unfounded, while at the same time much lime was removed, and a large quantity of phosphoric acid dissolved out by a 1 per cent citric acid solution. It was at last agreed that the new method should be thoroughly tested, both by the Biebrich chemist and by Dr. Wagner, and the results communicated to the London house as early as possible.

In October, after my return to Canada, I caused some trials to be made on basic slag, using the process above mentioned for removing free lime, and now desire to mention some of the results to this association. Three samples were experimented on, being Nos. 1128, 1146, and 1147 of Bulletin No. 70. Their contents in phosphoric acid, as claimed by the venders, are given first in the following table, and then the results of the successive treatments to which they were subjected.

Comparison of methods in determination of phosphoric acid in basic slag.

Sample No.	1128.	1146.	1147.	16774.	19336.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Total phosphoric acid claimed.....	17.00	18.32	18.18		
Available phosphoric acid claimed, determined by Wagner's process and 2 per cent solution.....		15.87	15.87		
Total phosphoric acid found.....	18.00	18.30	17.60	13.11	16.06
Available phosphoric acid, found by the ordinary United States citrate treatment.....	6.90	8.35	6.83	6.82	5.83
Lime (stated as CaO) dissolved out by boiling 5- gram sample for sixty minutes with 20 grams am- monium chlorid in 200 cc of water, replacing the loss of the latter; "A" treatment.....	7.53	7.01	8.06		
Lime dissolved by boiling 5-gram sample for thirty minutes with 5 grams chlorid ammonium in 100 cc water, replacing latter; "B" treatment.....	6.74	5.56	7.57		
Lime dissolved by boiling 5-gram sample for sixty minutes with 5 grams chlorid ammonium in 50 cc water, replacing latter; "C" treatment.....	8.18	5.76	8.29	6.52	6.43
Phosphoric acid remaining in residue from "A" after treating it with citrate of ammonia by the United States official method.....	6.33	6.91	7.16	Lost.	5.63
Consequently available phosphoric acid by the "A" treatment.....	11.67	11.39	10.84		10.43
Phosphoric acid remaining in the residue from "B," after occasional agitation for 30 minutes in the cold with 500 cc of 1 per cent citric acid solution.....	11.39	7.04	5.87		
Consequently available phosphoric acid by the "B" treatment.....	6.61	11.26	12.13		
Phosphoric acid remaining in the residue from "C," after energetic shaking in a horizontal mechanical agitator for 30 minutes, with 500 cc of 1 per cent citric-acid solution.....	4.60	3.20	3.58	3.45	2.69
Consequently available phosphoric acid by the "C" treatment.....	13.40	15.10	14.42	9.66	13.37
Recapitulation as regards available phosphoric acid without boiling in chlorid ammonium.....	6.90	8.35	6.83	6.82	5.83
By treatment "A".....	11.67	11.39	10.84	Lost.	10.43
By treatment "B".....	6.61	11.26	12.13		
By treatment "C".....	13.40	15.10	14.42	9.66	13.37
By Wagner method.....		15.87	15.87		

It will be observed from the experiments just described that from $5\frac{1}{2}$ to $8\frac{1}{4}$ per cent of lime were dissolved out of the samples by the ammonium-chlorid solution, and that this occasioned an average increase of nearly 4 per cent in the citrate soluble or available phosphoric acid as determined by the ordinary official citrate of ammonia method. When 200 cc of 1 per cent citric-acid solution is substituted for the citrate of ammonia (100 cc of a 20 per cent solution) and allowed to act in the cold with moderate agitation, the increase is about the same. When, however, vigorous mechanical agitation is employed the increase in the citric soluble or available amounts to 7 per cent. To me it does not appear reasonable that mechanical agitation should be used, that being so very unlike what obtains in nature.

The modification which I have to suggest in the official methods under "3. Determination of phosphoric acid (4) and (5)" on p. 13 of "Methods of analysis," Bulletin No. 46, revised edition, is as follows:

(4) Citric insoluble phosphoric acid.

(a) In acidulated samples: Introduce the filter containing the washed residue from the water-soluble phosphoric acid determination into a flask with 100 cc of 1 per cent citric acid solution. Stopper tightly and shake violently until the filter paper is reduced to a pulp. Add 100 cc additional of the 1 per cent citric-acid solution and digest in the cold for 30 minutes. Shake the flask every five minutes. Filter and wash thoroughly. Dry and transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed, add from 10 to 15 cc of strong hydrochloric acid and digest until all phosphate is dissolved; or return the filter with contents to a digestion flask, add from 30 to 35 cc of strong nitric acid and from 5 to 10 cc of strong hydrochloric acid and boil until all phosphate is dissolved. Dilute the solution to 200 cc, mix well, filter through a dry filter, take a definite portion of the filtrate, and proceed as under total phosphoric acid.

(b) In nonacidulated samples: In case a determination of citric-insoluble phosphoric acid is required in nonacidulated samples, such as basic slag, ground bone, bone ash, etc., it is to be made by taking 2 grams of the phosphatic material (without previous washing with water) and introducing it into a flask with 100 cc of a 5 per cent solution of chlorid of ammonium and boiling it for 30 minutes, replacing always the evaporated water; then filtering and washing the residue and treating it exactly as above described with 1 per cent citric acid solution. In case the substance contains much animal matter (bone, fish, etc.) the residue insoluble in citric acid is to be treated by one of the processes described under total phosphoric acid, a_2 , a_3 , or a_4 .

5. Citric-soluble phosphoric acid.

The sum of the water-soluble and the citric-insoluble subtracted from the total gives the citric-soluble phosphoric acid. The sum of the latter and the water-soluble phosphoric acid is to be regarded as "available" phosphoric acid.

I make this suggestion with all due deference only for what it is worth, and in order that this association may have a definite proposal before it in the event of its deciding to investigate the subject further. I would respectfully represent that the matter does deserve investigation at your hands, and I believe that if it is undertaken the result will be a decided step toward establishing such methods of analysis as will obtain acceptance not only in the United States but also in British countries and on the continent of Europe.

Mr. MYERS. I am very glad that Mr. Macfarlane has brought up this subject and offered some basis upon which this convention may take action. Some three months ago I was asked by a gentleman of the Thomas Phosphate Propaganda in Berlin to bring before this convention the question of the analysis of Thomas phosphate imported into this country. We are all aware that the phosphoric acid in Thomas slag is in a form that gradually becomes available when applied to lands of a certain character, whereas our methods of analysis in this country fail to show the actual value of the phosphoric acid present in the compound. In other words, our methods in this country are adapted for the analysis of acid phosphates and for such phosphates as are produced in our processes of manufacture here on a large scale, but are not adapted to this by-product of another manufacturing process, although the Thomas phosphate is a product that may come into this country on a large scale soon; and in fact is already coming in to a considerable extent at two or three points. There is none at all being received along the Atlantic coast, but large quan-

tities are used in Texas and California and the consumption is likely to increase on the Pacific coast. Those interested would like this association to take steps to improve their methods of analysis of this product. It is simply justice to them. They are not adulterating their goods, but the difficulty is in the processes of analysis. The function of the office of official chemist, as I conceive it, is not to prevent the sale of an article, but it is to prevent its adulteration. These goods, as a rule, are not adulterated, but our processes of analysis are such that they can not get the proper valuation, and they do not show their real worth. What they ask is that this association should take up this matter, investigate it, and settle upon some method of analysis which will be mutually satisfactory to the State chemists of this country and to the producers of this product. It is a reasonable request, and I am confident that the association will instruct its reporter next year, at least, to take up this question and go to the bottom of it so far as he can, and in the meantime make provision so that the chemists in the States of Texas and California and in Canada may have some means of properly controlling the inspection of these goods.

The PRESIDENT. Is there any further discussion?

Mr. HUSTON. I am quite a little interested in the method which Mr. Macfarlane has referred to with ammonium chlorid. It recalls my troubles of some years ago, for we have done a good deal of work with these materials in our laboratory. While I think it is quite right that the association should give reasonable attention to the request of a manufacturer of a product which does not seem to fulfill the manufacturer's expectations when tried by our methods, I am convinced that the Wagner method is utterly without value for working basic slag. Any reagent the curve of whose solvent action will go up rapidly and then return to zero is worthless for this purpose. With the Wagner reagent there is an enormous temperature range, and a very great difference in the amount dissolved according to the time involved. If you undertake to remove the free lime with ammonium chlorid you take out every element in the slag but phosphate. You can tear slag to pieces if you treat it with ammonium chlorid long enough. I much prefer sugar solution, as I think it disturbs the slag much less. I think we are putting ourselves to a needless trouble in trying to determine availability in basic slag. That is simply a question of total phosphoric acid and degree of fineness.

Mr. WHEELER. A large number of tests have shown very strong evidence that a 2 per cent citric-acid solution does give results according very closely with plant tests. It seems necessary to have immediately some method for the analysis of basic slag, and I think it would not be an unsafe policy to adopt the 2 per cent citric-acid solution until such time as the association can assure itself of a better procedure.

Would it be contrary to the constitution to adopt any method of analysis tentatively? Could this method be adopted as a provisional method for basic slag? Would it be constitutional?

The PRESIDENT (after reading section 7 of the constitution). I think it could be done constitutionally. If we had a method already we could not; but as we have none, we can adopt one.

Mr. WHEELER. I am interested in this matter because basic slag is of great importance in our agriculture. Some shipments were made to Rhode Island this year, and at any minute the goods are likely to be put on sale and it seems to me we must meet this problem at once. I think the gentlemen here are perfectly familiar with the fact that careful and extended tests have been made in Europe and we know the method adopted by the German chemists does agree very closely with plant experiments. I move that we adopt some method to use with basic slags temporarily. I move the adoption of the 2 per cent citric-acid solution method as a provisional method until such time as we are in a position to adopt a method as official.

Mr. MAGRUDER. I second Mr. Wheeler's motion. In Virginia we have basic slags to analyze, and I don't know what to do with them. I would like the association to adopt some method for them.

Mr. FREAR. I have experienced something of the same difficulty with this matter, and I shall be very glad to second the motion of Mr. Wheeler.

Mr. DYER. Perhaps you would like to know what we do on our side in regard to this matter. We have no official processes of fertilizer analyses in England, and in the matter of basic slags the commercial custom is to determine only the total phosphoric acid and take into account the degree of fineness. These two things together are taken as the measure of value. It is within my own personal knowledge that there are now on the market varieties of slag which are the result of a somewhat different process of steel making and having such variation that some have almost twice the availability of others. Mr. Macfarlane mentioned that when he was over there he referred this matter to our Society of Public Analysts, of which I have the honor to be president, and a committee was appointed to consider matters which Mr. Macfarlane brought before us. The committee consisted of those members who are more especially connected with fertilizer analysis. That committee made a report to the council of the society shortly before I sailed, and the council meeting at which this report was presented has taken place since I left England. I presume this report will be adopted and I think it will be interesting if I read to you the report of the committee on the question:

Resolution of the subcommittee appointed by the council of the Society of Public Analysts, London, England, which met on October 16, 1900, to consider Mr. Macfarlane's proposal relative to the analysis of fertilizers.

The committee have very carefully considered Mr. Macfarlane's letter and the documents therein referred to, but do not see their way to recommend the council to take steps in the direction either of laying down standards or of prescribing arbitrary methods for the analysis of fertilizers.

With regard to the subject more especially dealt with in Mr. Macfarlane's report (Bulletin No. 70), the committee do not make any recommendation in regard to the formulation of an official process for the estimation of available phosphoric acid in basic slag or other fertilizers. The committee, however, are unanimously of opinion that the ammonium citrate process, which is at present officially used in the United States of America, while affording useful means for the proximate determination of reverted phosphate in superphosphate, dissolved bones, and similar acid manures, is, nevertheless, in no sense an adequate means of measuring the amount of available phosphate that does not happen to have gone through the process of solution and reversion.

The committee are further of opinion that the citrate of ammonium process is wholly inapplicable to the analysis of basic slag, and that if any process of analysis is to be used for distinguishing between total and available phosphate in that manure it must be an acid process and one proceeding on some such lines as the present process of Professor Wagner.

This is a very cautious resolution, but it covers what is virtually a recommendation.

Mr. HILLS. I have not followed the discussion very closely, but there is one point about which I would like to speak. I believe our State laws require that the reverted phosphoric acid be dissolved with ammonium citrate.

Mr. WHEELER. Is there more than one State law in the Union that requires the determination to be made at a given temperature?

Mr. CARPENTER. I don't think it is generally recognized that basic slag is as valuable as acid phosphate, but if this acid test is applied, as I understand it, the result is that it is put on the same basis as acid phosphate. The results on some soils of basic slag may prove as beneficial as those produced by using acid phosphate, but on other soils the results with basic slag are of hardly any value at all, and it seems unjust to manufacturers of acid phosphate to put an article of this sort on the same basis.

Mr. MYERS. As I understand it, Mr. Huston struck the nail on the head when he said that the total phosphoric acid and the degree of fineness had a great deal to do with the question. You are dealing with a special compound in this case which is almost as distinct from a manufactured acid phosphate as sulphate of potash is. The question is whether there is in that compound any of the material which we call available phosphoric acid in the sense in which we use that term in describing an acid phosphate. I doubt exceedingly whether this is the case, but plant and pot experiments have demonstrated beyond doubt that when this material is mixed with the soil it does bring

results, and the action to make a tentative method of analysis for these goods seems to me to be an exceedingly wise one. Those gentlemen who have absolute laws regulating the methods of analysis in their States can apply them in such way as they see fit, but as a general preparation for the whole country it seems to me that there should be some tentative process for the determination of its constituents.

Mr. FREAR. When the day comes, and it seems to be coming now, that basic slag is brought into our land, it seems to me it would be wiser for us to follow those methods of analysis which have been adopted as the result of long experience abroad, so that our results will be comparable. In that way I think we will be able to avoid controversy with importers and manufacturers and dealers on the other side.

Mr. DYER. Though we have no official process at home, I often have to make analyses of slag sold on the Continent, and I can say of the Wagner process that it gives concordant results if worked with any reasonable degree of care, which is a good deal to say of an arbitrary process which dissolves only a fractional part of the substance. Different operators have not the slightest difficulty in getting concordant results.

Mr. MACFARLANE. Since the opinion of this meeting seems to be that Wagner's method should be adopted, I would like to ask if the gentlemen have considered the possible consequences. Suppose a manufacturer of superphosphate finds that this material is imported into your country and incorporated with a certain amount of phosphoric acid, and he finds also that you use this 2 per cent solution, and at the same time you apply a totally different solution to the insoluble part of his fertilizer. I think he is very likely to make objection. I know it to be the case that the fertilizer manufacturers in Canada would make very decided objections. I think it would justly cause controversy if you applied the Wagner system to the basic slag and not to other fertilizers. I have no doubt that Wagner's experiments were thorough and correct, and that in his agricultural experiments and experiments in his laboratory and garden he obtained results which justify him in saying that the proper solution is a 2 per cent solution. It seems to me that it is very necessary that you should be able to say to manufacturers, "You all get the same treatment." I would like to impress upon you the necessity of regarding the subject from the point of view of the manufacturers.

Mr. WHEELER. I ought to have stated that my motion was not made with the idea of putting off consideration of the paper presented by Mr. Macfarlane, because I had intended to move after this motion that the paper be referred to our committee on recommendations and be given due consideration with the idea that its suggestions might be taken up and tried by our association. But in the meantime we ought to have some method, and it seems to me that the 2 per cent solution is that one.

The PRESIDENT. You suggest that the paper be referred to the committee on recommendations. Would it be entirely proper that your motion should go to that committee also or to a special committee?

MR. WHEELER. I would move that the motion to adopt the 2 per cent citric acid solution, and also the consideration of the paper, be referred to our committee on recommendations.

Motion seconded.

MR. BARTLETT. Is not this going to give a chance to the manufacturers of bone meal, and other kinds of raw phosphates, to demand that this same method be used on their goods? It seems to me they could make that demand, and that until we know what method should be used in this country it would be better to act upon the suggestion of Mr. Huston and consider the fineness of the material and the total phosphoric acid it contains.

The motion of Mr. Wheeler was carried.

Mr. Williams then presented a paper as follows:

KILGORE'S MODIFICATION OF THE VOLUMETRIC METHOD OF ESTIMATING PHOSPHORIC ACID.

By C. B. WILLIAMS.

In the laboratories of fertilizer control stations and other institutions where a large number of determinations of phosphoric acid are required to be made quickly and accurately each year, it has been recognized for some time as almost imperative that some method shorter than the "gravimetric" should be devised. In 1894, Mr. Kilgore, then reporter on phosphoric acid for the Association of Official Agricultural Chemists, realizing this urgent demand, was the first to take up systematically the task by first thoroughly investigating the modified volumetric method himself, and then submitting it, as reporter, to the test of the association. In his report he gave credit to Mr. Henry Pemberton, who had the previous year published the description of a method based on this principle and which had been used very satisfactorily by a number of chemists. With this latter method Mr. Kilgore had obtained fairly good, but not uniformly good, results.

After considerable experimentation as regards precipitant, precipitation, and filtration, he proposed a modification of this method, stating that he had found that the modification gave him more satisfactory results with fertilizers. Since then each successive year has witnessed a still further modification, until now we have a method that is used in a large number of laboratories in America, with very gratifying results both in point of accuracy and rapidity.

By the adoption of the shaking method the writer was enabled, during the past spring, to precipitate, wash, and titrate, according to the modified volumetric method, 30 phosphoric-acid samples daily. The reagents used are the same as those prescribed for the volumetric method by the Association of Official Agricultural Chemists, except that the strengths of the standard solutions of potassium hydroxid¹ and nitric acid are made up so that 1 cc of each will represent 0.5 milligram of phosphoric acid, this being one-half the strength given in the association method.

¹ The standard potassium hydroxid is rid of CO₂ by first dissolving it in 95 per cent alcohol, letting settle, and then filtering off by reverse filtration the supernatant solution free from the insoluble potassium carbonate. This method is much quicker and simpler than the barium hydroxid method.

The method as carried out is as follows:

Total phosphoric acid is brought in solution in the usual way by boiling in a 200 cc flask, on a sand bath, 2 grams of fertilizer with 30 cc of concentrated nitric acid and 10 cc of concentrated hydrochloric acid to about 8 or 10 cc concentration; except in fertilizers containing much iron and alumina, in which instance 30 cc of concentrated hydrochloric acid alone is first added and boiled for about 30 or 40 minutes; then, after slightly cooling, 30 cc of concentrated nitric acid is added and the boiling continued until the excess of hydrochloric acid is removed. After cooling, make up to volume and take an aliquot part after filtration, or allow to stand several hours before measuring out. The latter is done in order that the supernatant liquid may become perfectly clear so it can be measured out with a pipette without filtration.

Twenty cc of solution, corresponding to 0.2 gram of fertilizer (except in samples containing over 20 per cent of phosphoric acid, when 10 cc is used), are measured into a 500 cc Erlenmeyer flask, the inside diameter of whose neck measures about 40 millimeters, and to it is added 10 to 12 grams of ammonium nitrate and 50 cc of distilled water. Neutralize the excess of acid with ammonia. When the contents have cooled, 30 cc of recently filtered molybdic solution are added and the flask, securely stopped with a rubber stopper, is placed in a Wagner shaking machine, which is revolved by a hot-air motor, and here shaken for 30 minutes. The shaking machine is maintained at 45 to 55 revolutions per minute, as this velocity has been found to give the maximum agitating efficiency. Remove flask from shaking machine and filter and wash by suction on a filter prepared as follows:

Through the rubber stopper in a 16-ounce pressure bottle of Erlenmeyer form is passed the small end of a carbon filter. In the bottom of this is a perforated porcelain plate or disk, to which is rigidly fastened a No. 19 copper wire, about 25 centimeters long, that projects downward into the pressure bottle. The disk is covered with a thin layer of asbestos.

After thoroughly transferring the ammonium phosphomolybdate and washing out the flask on to the asbestos filter, six more washings are given the precipitate. Then remove the stopper from the pressure flask with the small end of the carbon filter still stuck through it and hold upright over the sink and wash the outside free from acid with distilled water. Reverse the carbon filter into the mouth of the flask that originally contained the precipitate, still holding the small stem, and by means of the copper wire that extends beyond the small end of the carbon filter push out the disk, asbestos, and precipitate into the flask; wash disk and inside of carbon filter carefully and titrate, using a stirring rod about 30 centimeters long to thoroughly agitate during the operation.

In determining insolubles, 40 cc of solution, corresponding to 0.4 gram fertilizer, are taken. The precipitation, shaking, washing, and titrating are practically the same as with totals, except that little or no water is added in preparing for precipitation.

During the past spring 1,000 totals and 1,000 insolubles were made by the above method with not a single incomplete precipitation, the yellow precipitate always coming down in a granular form that was easily filtered and washed. Distilled water must be used in washing, as the suspended matter in ordinary water, in case it contains any, will not only retard filtration, but will form a compact coating over the precipitate that will greatly increase the difficulty of effecting a solution of the yellow precipitate with standard alkali as well as obscuring the color change of the indicator.

During the past year a large number of comparative results to test the volumetric method, as described above, with the regular official gravimetric method of the association, have been obtained in the laboratory of the division of chemistry, North Carolina department of agriculture, on commercial fertilizers offered for sale in the State. In all instances results were extremely satisfactory.

During the past summer three samples (two of ground phosphate rock and one of American slag) sent out by the referee on phosphoric acid for the Association of Official Agricultural Chemists were analyzed, with the following results:

Comparative determinations of phosphoric acid by gravimetric and volumetric methods.

No.	Gravi- metric method.	Volu- metric method.	No.	Gravi- metric method.	Volu- metric method.
	<i>Per ct. P_2O_5.</i>	<i>Per ct. P_2O_5.</i>		<i>Per ct. P_2O_5.</i>	<i>Per ct. P_2O_5.</i>
389.....	13.55	13.45	390.....	17.35	16.90
	13.43	13.50		17.33	
		13.51		17.27	
		13.50	391.....	26.01	25.90
390.....	17.21	16.88		26.10	25.80
	17.23	16.93		26.11	25.85
	17.27	16.95		26.02	25.88

It will be noticed that the gravimetric results on sample No. 390 are perceptibly higher than those determined volumetrically. This is most probably due to the presence of iron in the magnesium pyrophosphate as a qualitative test, for iron revealed its presence there. As sample No. 391 contains 4.70 per cent of ferric oxid, this may account for the wider variation than in No. 389 between the gravimetric and volumetric results.

Mr. BARTLETT. Before leaving the subject of phosphoric acid, I would like to say a word. We were unable to cooperate in this work during the past year because of the pressure of other matters. By a strange coincidence, the work in which we were engaged, namely, the determination of alumina in baking powder, is found to bear directly on the subject under discussion. This may seem strange, but as a matter of fact many of the baking powders on the market contain the same mineral ingredients as basic slags, although in different proportions. The percentage of alumina is the same, ranging from 3 to 6 per cent. The method which we have used, and quite successfully, is the acetate method. I would suggest that while this matter of the determination of alumina, iron, phosphoric acid, and lime is being considered in the case of fertilizer materials it might be well to consider the same bodies as found in baking powders. If these two matters can be taken up together I think the work of the association would be facilitated. The acetate method, in a somewhat different form, has given results which agree closely with the old standard method. This may be out of order, but I do not think it would be in order in connection with the examination of food products.

After some discussion as to the time of holding the next session, the meeting adjourned to meet at 9 o'clock the following morning.

SECOND DAY.

SATURDAY—MORNING SESSION.

The meeting was called to order at 9 a. m., with the president, Mr. Kilgore, in the chair.

The report on sugar was then called for, and the following letter from the absent referee was read:

SAN FRANCISCO, CAL.,
October 25, 1900.

Dr. H. W. WILEY,
Secretary Association of Official Agricultural Chemists,
Washington, D. C.

DEAR SIR: Owing to my departure for the Hawaiian Islands, I regret to inform you that I have not been able to complete the report of the referee on sugar in time for the meeting of the association in November. The work preliminary to my leaving the sugar experiment station has so occupied my time as to render it impossible to finish my report as referee.

The amount of cooperation received in this work has been very small, and only two reports have been received, one from Dr. John White and the other from Mr. R. B. Hellner, both of Nebraska. These, together with the work done at the sugar experiment station in Louisiana, are all the data at hand, and from this very few conclusions can be drawn. I hope that the absence of the report this year will not detract from the association work.

I also sincerely regret not being able to comply with your request regarding the revision of Bulletin No. 46, but the same reasons apply in this instance.

Again assuring you of my regrets, etc., and hoping for a successful and instructive session of the association, I am,

Yours very truly,

R. E. BLOUIN,
Referee on Sugar.

Mr. WILEY. I thought the members might be interested in the brief report of the international committee for the unification of methods of sugar analysis at the third international congress of applied chemistry, which was held in Paris in August, 1900. This committee was appointed by the international congress of applied chemistry at the Paris meeting in 1896. The first meeting, which I did not attend, was held at Braunschweig. The second meeting was held in Vienna, in 1898, with about thirty members of the committee and invited guests present, under the presidency of Dr. Herzfeld, of Berlin. Twelve members of the committee were present at the Paris meeting, also under the presidency of Dr. Herzfeld.

The chief points discussed were the influence of temperature upon the specific rotation of sugar and the adoption of general principles of analysis for international guidance.

After a prolonged discussion the committee voted to recommend that all polariscopes be standardized for the usual temperature at which they were employed, the committee recognizing the fact that

variations in temperature caused marked changes in specific rotation. This principle is one which has been put in practical operation in this country, in official work, for more than ten years. It has also been recognized in the British West Indies for a longer period and proper corrections made in the polarizations for the high temperatures which there prevail.

The specific rotation of sugar decreases as the temperature rises, while the specific rotation of quartz increases with a rising temperature. Since in ordinary instruments the compensation for the sugar solution is secured by means of a quartz wedge, a less thickness of it is required for a given degree of rotation at a high temperature than at a lower one. Thus the reading of the scale, which is attached to the quartz wedge, is lower than it really should be. At the same time the specific rotation of the sugar diminishes, and thus adds to the error.

Most careful measurements of the magnitude of these errors, on quartz compensating instruments, have shown that they amount to about one-tenth of a per cent of sugar on the sugar scale for each rise of 3° of temperature. Since the average temperature of polarizations in this country is nearly 10° higher than the temperature at which our standard instruments are graduated, viz, 17.5° , it is seen that the average magnitude of the error in this country would be almost three-tenths of 1 per cent.

The importance of allowing for this error, both in commercial valuations and in the classification of sugars for customs duties, is at once apparent. It is gratifying to know that the international committee on the unification of sugar analysis has accepted the principle of correction which has been adopted for so many years in the official work of this country.

In experiments conducted in my own laboratory I demonstrated beyond doubt the magnitude of these errors, and the paper containing the results of my investigations was presented at the meeting of the international congress of applied chemistry and also published in the *Journal of the American Chemical Society* for July, 1899.

MR. FRAPS. I would like to ask a question as to the temperature at which these experiments were carried out, and what kind of sugars were used.

MR. WILEY. The highest temperature which I used in my work was 40° and the lowest was zero.

MR. FRAPS. What kind of sugar did you use?

MR. WILEY. Chemically pure sugar.

The report on soils was then presented by Mr. Hartwell.

REPORT ON SOILS.

By BURT L. HARTWELL, *Referee*.

As the analytical data included in the present report represent the work of two years, it may not be out of place to review briefly what has been done since my appointment as referee on soil analysis in 1898.

As soon as possible after that time a circular letter, published in our last proceedings, was sent to the Experiment Station chemists. This letter stated the recommendations made by this association at the meeting held here two years ago regarding the soil work. They were (1) a trial of the so-called international method for determining assimilable potash, i. e., using dilute nitric acid as a solvent; (2) a further trial of Hollemann's method for the determination of the active lime compounds, i. e., using water saturated with carbonic acid, and (3) further tests with alkaline ammonium chlorid as a solvent for potash. An appeal was made for suitable soils for carrying on the work of testing solvents which should serve for the determination of the more active soil constituents. In another paper will be presented the results of some preliminary work upon the soils received in response to this request. Suggestions concerning the work and cooperation were also urged.

It was deemed advisable, on account of the immediate need for samples, to distribute to those who expressed a willingness to take part in the investigations certain soils upon which considerable work had already been done by this association, and in addition two soils from California. It has been thought best to present in this connection such information as it has been possible to obtain concerning the soils employed.

DESCRIPTION OF THE SOILS.

No. 1, from Junction City, Boyle County, Ky., farm of Thomas R. Walker.—From the same lot of soil as was distributed as No. 1 by the referee for 1898. A light clayey loam, cleared from original timber about twenty-five years ago. Probably of the Devonian formation. Surface soil 4 to 6 inches deep. Below this is a yellow clay subsoil mixed with gravel, 2 to 4 feet in depth, and under this latter a blue clay.

Field experiments were conducted under the direction of the Kentucky Experiment Station upon land which this sample represents, the experiment with potatoes being upon the same field from which the sample was taken, and those with maize and beans upon the same kind of land. Below are given the yields per acre:

Yield per acre of potatoes and ear corn, 1895-'96.

No. of plat.	Fertilizers supplied.	1895, potatoes.	1896, potatoes.	1896, ear corn.
		<i>Bushels.</i>	<i>Bushels.</i>	<i>Bushels.</i>
1, 5, 10, av ..	None.....	78	46	14
2.....	N	72	45	10
3.....	P ₂ O ₅	122	155	25
4.....	K ₂ O	77	55	14
6.....	N, P ₂ O ₅	146	208	37
7.....	N, K ₂ O	75	71	15
8.....	P ₂ O ₅ , K ₂ O.....	125	171	28
9.....	N, P ₂ O ₅ , K ₂ O	121	284	27

The fertilization per acre was 160 pounds of nitrate of soda, 140 pounds of double superphosphate, and 160 pounds of sulphate of potash.

Navy beans, 1896.

Fertilization.	Crop in pounds.
None	909
750 pounds Orchilla guano	1, 134
750 pounds acid phosphate	1, 620

The sample was taken to the depth of 4 to 6 inches, in March, 1898, from many places. The land had received an application of 200 pounds of acid phosphate per acre a few months before. It is impossible to say how much influence this application would have upon the amount of phosphoric acid removed by weak acids, but even if all of the available phosphoric acid in the acid phosphate should be removed by the weak acid solvents, which is not at all probable, the percentage would not be increased 0.003 per cent. The entire amount of phosphoric acid actually found by treatment with N/5 hydrochloric acid within a year after the application of acid phosphate was made amounted to only 0.0026 per cent.

Both crop records and analysis showed this soil to be deficient in assimilable phosphoric acid, and it was again distributed in 1899 chiefly for the determination of the amount of phosphoric acid soluble in N/5 nitric acid. This solvent was recommended for trial by the association for determining active potash, and it was thought desirable to ascertain if its action upon the phosphoric acid of the soil were similar to that of N/5 hydrochloric acid, which had been adapted provisionally; if so, a weak solution of nitric acid may be applicable for the determination of the more active forms of both phosphoric acid and potash, and thus make the use of two solutions unnecessary.

No. 2. From the Kentucky Agricultural Experiment Station.—From the same lot of soil distributed as No. 2 by the referee for 1898. The soil was taken in March, 1898, from acre P., plat 7, at the depth of 5 or 6 inches. The land had never received any fertilizer, and experiments carried on for a number of years indicate a marked deficiency in available potash.

A description of this soil, the crop records, and analytical results heretofore obtained upon the same by this association are given in my last report.¹ This soil was distributed for the estimation of potash soluble in N/5 nitric acid and in alkaline ammonium chlorid.

No. 3. From the Rhode Island Agricultural Experiment Station.—This soil was sampled to the depth of 4 inches in the fall of 1894 from the unfertilized plot of a soil test. Samples collected at the same time from plots 2 and 5 of this experiment were used by the referee for 1895. A description of the soil, crop results, and analytical data may be found in the proceedings for 1899 (p. 81). The soil needs liming or other alkaline treatment to insure a good yield of most crops. It was distributed for the determination of lime soluble in N/10 acetic acid and in water saturated with carbon dioxide.

No. 4. From Ventura County, Cal.—Orange orchard of N. B. Blanchard near Santa Paula; a representative of the arid upland or bench soils. A silty soil of light umber color when dry, becoming blackish when wet; very easily tilled, retaining its tilth remarkably, so that an ax handle can be thrust perpendicularly into it the entire length with little exertion. In the lower bench of the valley where the sample was taken the material remains apparently the same to a depth of from 12 to 20 feet; toward the hills there is a second bench where the soil is apparently the same, but of a slightly reddish tint; on the mountain slopes the soil, still quite similar in its working qualities, is of a decidedly reddish tint and is remarkable for its retention

¹ Proceedings A. O. A. C., 1899, p. 78.

of natural moisture, enabling it to produce corn without irrigation. This soil was distributed as one containing plenty of plant food, and the various determinations by the use of weak solvents were requested for comparison with results obtained on the deficient soils.

No. 5. From the Sierra Foothill Station, near Jackson, Amador County, Cal.—Sample taken to the depth of 12 inches from about half way down the central hill; an orange-red loam, the lumps of which are easily crushed between the fingers when dry, and show considerable coarse sand; when wet it becomes only moderately adhesive, while its color darkens materially; slate fragments are intermingled more or less, much of the sand being comminuted slate. The natural growth¹ characteristic of this soil includes all the trees and shrubs of the foothills at large, so that as far as the vegetation can indicate, this red soil is representative of the higher class of land on which the poison oak and toyon alone are found in the uplands. This soil contains a high percentage of calcium and potassium oxids, which render soils of this character very productive for a while, even though the amount of phosphoric acid is small. Farmers have, however, after a time found it to their advantage to supply phosphoric acid. It was thought desirable to test the solvents upon this soil, which is representative of so large a class.

Directions for the work to be undertaken upon the above-mentioned soils were distributed in March, 1899. It was possible to secure so few results before the meeting of the Association of Official Agricultural Chemists at San Francisco in July, 1899, that the cooperating chemists were requested to continue the work with the same soils and directions during the present year. These directions were unexpectedly published in connection with my last report. They contain a few mistakes which should be noted, viz, next to the last line in section 2 reads, "and 5 cc strong nitric acid," the word "and" should be replaced by "add;" soil No. 5 was published as coming from Arlington Heights, southern California. This could not be obtained in time, and a soil from the Foothill Station, as previously mentioned, was finally employed as No. 5.

MECHANICAL ANALYSES.

Mechanical analyses of soils from plats in close proximity to those from which Nos. 2 and 3 were taken may be found on pages 79 and 81, respectively, of the proceedings for 1899. Below are given results furnished by Loughridge, of Berkeley, Cal., which represent the mechanical condition of the soils from the localities where Nos. 4 and 5 were taken.

Mechanical condition of soils from same localities as samples Nos. 4 and 5.

Soil.	Similar to No. 4.	Similar to No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>
Gravel over 1.2 mm diameter.....		13.95
Gravel between 1.2 and 1 mm diameter		.20
Coarse sand between 1 and 0.6 mm diameter.....	3.53	5.85
Fine earth less than 0.6 mm diameter	96.47	80.00
	100.00	100.00

¹Cal. Agr. Expt. Sta. Rpt., 1888 and 1889, p. 79.

Hydraulic values of the fine earth borne up by water rising at the velocities indicated in millimeters per second.

Constituents of fine earths.	Hydraulic values.	No. 4.	No. 5.
	mm.	Per cent.	Per cent.
Sand	64	4.11	2.17
	32	6.45	2.89
	16	7.01	5.04
	8	11.48	4.03
	4	16.36	5.91
Silt	2	12.67	8.21
	1	8.03	7.68
	.5	5.14	9.96
	.25	1.20	4.64
	.25	14.04	30.26
Colloid clay	.0023	15.02	16.43
Hygroscopic moisture in fine earth.....		101.51	97.22
Water-holding capacity		5.49	6.00
		32.10	47.50

Soils Nos. 1 and 2 had been passed through a sieve approximately 1 mm in diameter, as was the case in 1898; No. 3 through a sieve about 2 mm in diameter; Nos. 4 and 5 through a 0.6 mm sieve.

Below are given the data furnished by A. M. Peter, of Kentucky, concerning the coarse soil of the lots from which Nos. 1 and 2 were taken:

Mechanical condition of coarse portion of soils from which samples Nos. 1 and 2 were taken.

Material.	No. 1.	No. 2.
	Per cent.	Per cent.
Larger than 3 mm sieve	1.69	0.67
Between 2 and 3 mm sieve.....	.73	1.05
Between 1 and 2 mm sieve.....	1.26	4.21
Between one-half and 1 mm sieve	.10	1.03

Soil No. 3 contained 95.55 per cent of material finer than 2 mm, 1.48 per cent between 2 and 4 mm, and 2.97 per cent coarser than 4 mm.

RELATIVE WEIGHTS OF THE SOILS.

The "apparent specific gravity" of Nos. 1 and 2 were obtained by Mr. Peter by packing the soil into a receptacle as tightly as possible, by jarring, and comparing the weight with that of an equal volume of water. The specific gravity for No. 1, with 1.01 per cent of moisture, was found to be 1.27 in the sifted and 1.23 in the unsifted soil; that for No. 2, with 2.16 per cent of moisture, was 1.25 for the sifted and 1.20 for the unsifted soil. Soil No. 3, air dried, when well shaken down, weighed 1.21 times as much as the same volume of water. Similar determinations were made upon the air-dried samples, Nos. 4 and 5, before sifting; the specific gravity of No. 4, with 2.14 per cent moisture, was 1.35, and of No. 5, with 1.12 per cent moisture, 1.19.

Although these data are not as complete in all cases as they should be, they enable one to calculate roughly the number of pounds of the dry soil which would be contained in an acre of land to the depths sampled. The number of pounds per acre in round figures is thus found to be for No. 1, at 6 inches deep, 1,590,000; for No. 2, at

6 inches deep, 1,500,000; for No. 3, at 4 inches deep, 1,010,000; for No. 4, at 12 inches deep, 3,470,000; for No. 5, at 12 inches deep, 2,560,000. These figures enable anyone to calculate roughly the amount of assimilable plant food per acre at the various depths from the percentages of the same which are found by analysis.

It must be plain to the members of this association that our percentage results upon agricultural soils of different depths should not be expected necessarily to be in direct relation to the crop-producing qualities of the same. A comparison of the amounts of a given ingredient found by analysis to exist in an assimilable form within a given area at the depth to which the roots penetrate would seem to be more reasonable. This necessitates a consideration of the depth to which the roots of the same kind of plant will seek plant food under the existence of different conditions. In making comparisons of the percentages of given ingredients found in the soils employed in the work of the present year these considerations should not be overlooked, particularly as there will be occasion to compare shallow soils from humid sections with deep soils subjected to different conditions as to rainfall.

COMPLETE ANALYSES.

The following determinations were made by Mr. McGuigan, Agricultural College, North Dakota.

Determinations made with samples Nos. 1, 2, 3, 4, and 5.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture.....	0.69	1.55	1.24	1.10	0.76
Volatile matter.....	5.26	7.66	9.54	2.83	5.71
Total silica (SiO ₂).....	87.49	78.40	81.51	87.39	78.15
Iron (Fe ₂ O ₃).....	4.50	8.00	4.10	3.58	5.64
Alumina (Al ₂ O ₃).....	0.87	2.39	2.01	2.44	7.88
Phosphoric acid (P ₂ O ₅).....	0.17	0.35	0.13	0.21	0.12
Manganese (MnO).....			0.08	0.09	
Lime (CaO).....	0.25	0.46	0.34	0.71	0.48
Magnesia (MgO).....	0.16	0.23	0.08	0.10	0.27
Sulphuric acid (SO ₃).....			0.29	0.12	0.07
Potash (K ₂ O).....	0.17	0.23	0.21	0.38	0.46
Soda (Na ₂ O).....	0.20	0.24	0.27	0.34	0.32
Total.....	99.76	99.51	99.80	99.29	99.86
Nitrogen.....	0.12	0.17	0.236	0.0623	0.07
Extractives (Humates).....	4.98	7.86	5.76	0.85	
Humus.....	2.00	3.02	4.34	0.22	
Alkalies.....	0.113	0.345	0.546		
Phosphoric acid.....	0.026	0.062	0.036		

The following statements from Mr. Ladd, of North Dakota, accompanying the above analyses, are explanatory of certain determinations, viz: "I have termed humates the entire extractives dissolved out by the ammonia solution after the soil has been treated with weak hydrochloric acid, as found by evaporating an aliquot portion of the extract. The phosphoric acid and alkalies reported are those found in this residue. The alkalies in the humus extract are weighed as chlorids."

POTASH.

While N/5 hydrochloric acid has been accepted provisionally as a solvent for assimilable phosphoric acid, we have not found any solvent for potash which has proved satisfactory in the minds of the referees. Recommendations to discontinue the use

of citric acid, calcium chlorid, and ammonium chlorid have been made by different referees. Further trial with the last solvent, however, was urged, and a method heretofore untried by the association recommended, viz, the so-called international method for available potash, in which dilute nitric acid is used. At the congress of chemists held in Paris in 1889 it was pointed out that with a 0.05 to 0.1 per cent nitric-acid solution, the quantity of potash which goes into solution increases by continued stirring of the mixture with the time of action of the acid up to a certain maximum which is reached in from three to four hours, and after that it is not changed even when the strength of the acid mixture is increased to 2 per cent.¹ If this be true a decided advantage at once appears in the use of dilute nitric acid. N/5 nitric acid was the strength determined upon by your referee that we might have a solution of definite normality and that the results obtained by the same solvent upon phosphoric acid would be comparable with those by N/5 hydrochloric acid, which has been adopted provisionally.

Thorn Smith stated in a letter accompanying his results that he lacked confidence in them because of constant unavoidable interruptions, in consequence of which the directions in many cases were not followed as to shaking and time of digestion.

C. C. Moore, instead of shaking the solution every half hour, as stated in the directions, used a continuous shaking machine. Having recorded these exceptions to the regular method as outlined, it has been thought best to include the results by Smith and Moore with all other results which follow without further comment. All of the succeeding results represent percentages of water-free soil.

Determination of potassium oxid in soil samples.

Analyst.	Solvent. <i>a</i>	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.	Soil No. 5.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
G. S. Fraps, Raleigh, N. C.	N/5HNO ₃		.0063			
Thorn Smith, Moscow, Idaho....			.0060		.0295	.0195
R. J. Davidson and W. E. Ellett, Blacksburg, Va.			.0087			.0178
J. A. Bizzell, Raleigh, N. C.					.0143	{ .0157 .0125
C. C. Moore, Division of Chem- istry, Washington, D. C.	N/5HCl	.0190	.0163	.0140	.0358	
M. E. Jaffa, Berkeley, Cal.		.020	.016	.019	.036	.029
Do		.010	.008	.012	.018	.013
Thorn Smith, Moscow, Idaho....	2 per cent citric acid. <i>b</i>	.0122				
Davidson and Ellett, Blacks- burg, Va.			.0075			
J. A. Bizzell, Raleigh, N. C.					.0151	.0139

a In the acid solvents allowance was made for the neutralizing action of the soil, so that the strengths at the end of the digestion are given.

b Fifty gms. of soil digested at room temperature in 300 cc of solution for twenty-four hours with frequent shaking.

¹ Wiley's Principles and Practice of Agricultural Analysis, p. 381.

The comparative yields from certain plats of field soil tests which furnished data as to the amount of assimilable potash.

The first three terms of proportions, the last terms of which are given in the following columns.	Soil No. 1, potatoes. Sampled in Mar., 1898.		Soil No. 2, corn on the ear. Sampled in Mar., 1898.		Soil No. 3, corn and stover. Sampled in fall of 1894.	
	1885.	1896.	1889-92.	1894-97.	1890, 1891.	1892, 1893.
Nothing plat; K ₂ O plat; :1:	0.9	1.2	1.8	1.9	-----	-----
N plat; N, K ₂ O plat; :1:	1.0	1.6	2.0	1.8	1.6	1.7
P ₂ O ₅ plat; P ₂ O ₅ , K ₂ O plat; :1:	1.0	1.1	1.9	1.9	1.3	1.4
N, P ₂ O ₅ plat; N, P ₂ O ₅ , K ₂ O plat; :1:	0.8	1.4	2.0	2.1	-----	-----

NOTE.—It will be evident from the above table that numbers greater than 1 show an increased yield from the use of potash, while those equal to or less than 1 show no increase.

According to the above table of comparative yields on the deficient soils, Nos. 1, 2, and 3, one would attribute the greatest deficiency of assimilable potash to No. 2; it is more uncertain, however, considering the different crops employed, as to whether No. 1 or No. 3 ranks next in deficiency. In view of the fact that there was no deficiency of assimilable potash in soil No. 1 during 1895, whereas there was a lack in soil No. 3 from the very first, it may be justifiable to conclude that the latter is more deficient. If these ideas be correct regarding the relative deficiencies of the three soils, then Jaffa's results with N/5 hydrochloric acid are in accord. The results by Moore with N/5 nitric acid are not in accord, because the least potash was not obtained from soil No. 2, which apparently was the most deficient. It will be noticed that no two of the acids agree concerning the relative amounts of assimilable potash in soils Nos. 1, 2, and 3 in the cases where comparisons have been possible. No conclusions, however, should be drawn from so few determinations, particularly when ideas concerning relative crop-producing powers are so liable to error.

All of the solvents employed for assimilable potash dissolved less potash from the deficient soils than from the fertile California soils. The same is seen to be true also of strong hydrochloric acid by glancing at the results by Mr. McGuigan given previously. The fact that only 0.001 per cent more of potash was extracted by 2 per cent citric acid from soil No. 5, which is supposed to be rich in assimilable potash, than from the deficient soil No. 3 should not be overlooked.

PHOSPHORIC ACID.

Phosphoric acid extracted by N/5 hydrochloric and nitric acids.

Analyst.	Solvent.	Soil No. 1.	Soil No. 2.	Soil No. 3.	Soil No. 4.	Soil No. 5.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
G. S. Fraps, Raleigh, N. C.	N/5 HCl.....	0.0026	-----	-----	-----	-----
	N/5 HNO ₃	.0029	-----	-----	-----	-----
J. A. Bizzell, Raleigh, N. C.	N/5 HCl.....	-----	-----	-----	-----	-----
	N/5 HNO ₃	-----	-----	-----	0.1341	0.0020
Thorn Smith, Moscow, Idaho....	N/5 HCl.....	-----	-----	-----	.1422	.0011
	N/5 HNO ₃	.0029	-----	-----	.1321	.0018
Davidson and Ellett, Blacksburg, Va	N/5 HCl.....	-----	-----	0.0284	-----	Tr.
	N/5 HNO ₃	.0019	-----	.0186	-----	Tr.
C. C. Moore, Division of Chemistry, Washington, D. C.	N/5 HCl.....	.0016	0.0666	.0265	.1350	-----
	N/5 HNO ₃	.0010	.0854	.0190	.1737	-----

Comparative yields from certain plats of field soil tests which furnished data as to the amount of assimilable phosphoric acid.

The first three terms of proportions, the last terms of which are given in the following columns:	Soil No. 1, potatoes sampled in Mar., 1898.		Soil No. 2, corn on the ear sampled in Mar., 1898.		Soil No. 3, corn and stover sampled in fall of 1894.		
					Unlimed.		Limed in 1896. ¹
	1895.	1896.	1889 to 1892.	1891 to 1897.	1890 and 1891.	1892 and 1893.	1898 to 1900.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Nothing plat: P ₂ O ₅ plat : : 1 : ..	1.6	3.4	0.9	1.0	-----	-----	-----
N plat: N, P ₂ O ₅ plat : : 1 :	2.0	4.6	1.0	0.9	1.7	3.0	1.5
K ₂ O plat: K ₂ O, P ₂ O ₅ plat : : 1 : ..	1.6	3.1	1.0	1.0	1.1	2.3	1.2
N, K ₂ O plat: N, K ₂ O, P ₂ O ₅ plat : : 1 :	1.6	4.0	1.0	1.0	-----	-----	-----

¹ Liming this soil has rendered the phosphoric acid less deficient in spite of the fact that under ordinary conditions the deficiency would be expected to become greater the longer the experiment is continued.

It will be evident from the above table that numbers greater than 1 show an increase in yield from the use of phosphoric acid, while those equal to or less than 1 show no increase. Comparisons of the percentages of phosphoric acid dissolved by N/5 hydrochloric and nitric acids show fairly concordant results in most instances. The same chemist extracts by one acid more or less than by the other, depending upon the sample of soil. In the case of a given soil, No. 4, one chemist obtains the most phosphoric acid by extracting with hydrochloric acid, while another obtains the most with nitric acid. Mr. Peter determined the phosphoric acid dissolved by N/5 hydrochloric and nitric acids from the soils distributed by the referee for 1897, and found, respectively, 0.0039 and 0.003 in the case of one soil, and in another 0.0357 and 0.029.

From the data thus far obtained one would say that any indications of the amount of assimilable phosphoric acid which the weak hydrochloric acid solvent might furnish would be afforded equally well by the same strength of nitric acid. Therefore, if N/5 nitric acid should prove to be a valuable solvent for determining relative amounts of assimilable potash in soils there would seem to be no reason, so far as the present results indicate, why it may not replace hydrochloric acid for the determination of assimilable phosphoric acid and thus secure the unquestionable advantage of making both determinations from a single solution. It should be noticed that less phosphoric acid was extracted from No. 3 by nitric than by hydrochloric acid; according to the crop results the nitric acid afforded the best indication of the amount of assimilable phosphoric acid in this soil. The percentage of phosphoric acid from Nos. 1, 2, 3, and 4 has increased in the direction of the increase in crop production as influenced by the same ingredient. According to the analytical results the California soil, No. 5, contains no greater percentage of assimilable phosphoric acid than the most deficient soil, No. 1. This does not agree with the impression which was received concerning the relative productiveness of these two soils, but in the absence of a soil test conducted upon the California soil in a similar manner as that upon soil No. 1 the writer is not inclined to conclude that this is an instance where our provisional method for assimilable phosphoric acid has entirely failed us. Upon a previous page it was stated that farmers had after a time found it to their advantage to supply phosphoric acid to soil represented by No. 5. A soil test conducted in pots upon lots of soil truly represented by the samples under discussion would be desirable before making a definite statement regarding the relative amounts of assimilable phosphoric acid in equal weights of these soils. Certain observations to be presented in another paper lead to this opinion. If it be con-

ceded for the time being that the crops drew their plant food from soil equal in depth to that at which the two samples, Nos. 1 and 5, were taken, viz, about 6 to 12 inches, respectively, there would be at their disposal per acre in soil No. 1, 31.8 pounds of assimilable phosphoric acid, and in soil No. 5, 51.2 pounds. These quantities are obtained by accepting 0.002 as the average percentage of phosphoric acid obtained by extracting with N/5 acid from each soil and multiplying by the weights of the soils per acre at the depths sampled, given on a preceding page. An excellent illustration is here afforded of the different conclusions which might be drawn depending upon the point of view. If the percentages only were taken into consideration it might be said that the amount of phosphoric acid at the disposal of the plants was the same in both soils, but when the weight of surface soil is also taken into account there is a great deal more phosphoric acid for the crop in soil No. 5 than in soil No. 1. According to the former way of looking at the matter our provisional method would be pronounced unreliable, but from the latter standpoint it would be said that the method gave results furnishing indications according in a general way with the crop records. It is hoped that this illustration may discourage the custom often followed of considering the percentages only of a given ingredient found by the analysis of two soils of different specific gravities, depths, and humidity. Many factors must be considered before a method which has often proved useful should be abandoned.

LIME.

Calcium oxid extracted by N/10 acetic acid.

Analyst.	Soil No. 3, deficient in calcium carbonate.	Soil No. 4, abundance of calcium carbonate.	Soil No. 5, abundance of calcium carbonate.
Thorn Smith, Moscow, Idaho.....	0.0209	0.1248	0.1030
Davidson and Ellett, Blacksburg, Va.....	.0068		.0906
B. L. Hartwell, Kingston, R. I.....	.0166		

According to the above results, dilute acetic acid indicates very well the difference in the content of the more active lime compounds which exists between the Rhode Island sample and those from California.

The directions for the soil work included the description of a device for maintaining the saturation of carbonated water throughout the entire time during which the soil was to be digested. By this means fresh carbon dioxid could be readily admitted whenever the strength of the solution had been lessened by its action upon the soil, and a maximum dissolving effect be exerted. The ease in thus maintaining a constant strength gives to carbonated water an advantage not possessed by other weak solvents. It is to be regretted that no results have been reported upon the use of water kept saturated with carbon dioxid.

HUMUS.

Determination of humus in soil samples Nos. 3, 4, 5.

Analyst.	Soil No. 3.	Soil No. 4.	Soil No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. A. Bizzell, Raleigh, N. C.....		1.56	2.17
Davidson and Ellett, Blacksburg, Va.....	3.58		
B. L. Hartwell, Kingston, R. I.....	4.00		
Thorn Smith, Moscow, Idaho.....	3.99	1.59	

The principal object in requesting determinations of humus was to see if there would be a satisfactory concordance between the results from different chemists. The results upon soil No. 3 show that some difficulty exists in obtaining parallel results under varying circumstances. Davidson writes: "The per cent of humus found will depend very largely on the amount of extraction with 1 per cent hydrochloric acid solution. The more you extract the less humus you get. As an experiment we ran one determination using about 500 cc of 1 per cent hydrochloric acid, and the per cent of humus from this was 3.14." The writer also found that by continuing the extraction with hydrochloric acid for some time after there ceased to be a reaction for lime, varying results were obtained, depending upon the amount of the extraction with hydrochloric acid, viz, 3.28 per cent, 2.88 per cent, and 2.77 per cent. The result published in the table above was an average of 3.98 and 4.01 per cent, which were obtained by running two tests side by side and stopping the extraction with hydrochloric acid as soon as the lime was removed. Emery¹ and Rimbach,² in their articles on humus, point out the fact that after the ammonia extract is evaporated and dried at 100° a certain amount of ammonia is retained by the residue which, according to the official method, would pass off upon ignition and cause a plus error. Rimbach ascertained the extent of this error by previously boiling another aliquot part of the ammonia extract to expel free ammonia and then distilling the residue with magnesia. The quantity found by the latter process was subtracted from the loss of weight by ignition in obtaining the true humus. Rimbach also concludes that by leaching with hydrochloric acid and water a certain quantity of humus, varying in the different soils, is lost to the subsequent extraction with ammonia. Snyder³ calls attention to the fact that certain soils give up some of the phosphoric acid to the dilute hydrochloric acid which otherwise would have been included in the ammonia extract. The above considerations suggest the possibility that the long extractions with hydrochloric acid may have removed from soil No. 3 certain ingredients which otherwise would have been extracted by the ammonia, and thus have increased the amount of so-called humus either directly or by virtue of having retained ammonia which could not be driven out at 100°.

ASPARTIC ACID METHOD.

This method, which was brought before the association in 1898 by Mr. Maxwell, of the Hawaiian Experiment Station,⁴ consists essentially in digesting the soil twenty-four hours with a 1 per cent aspartic acid solution, shaking gently every fifteen minutes during the daytime. At the request of the referee Mr. Maxwell expressed a willingness to test the soils which have been under consideration. Unfortunately only one of the samples, the arid soil from California, No. 4, was analyzed. The following percentages were reported: Calcium oxid, 0.1334; potassium oxid, 0.0170; phosphoric acid, 0.0294. For comparison with these amounts the average results from 30 Hawaiian soils were also reported, as follows: Calcium oxid, 0.1400; potassium oxid, 0.0406; phosphoric acid, 0.0025. The following is quoted from Mr. Maxwell's letter of June, 1899: "These results justify the warning statement made by me a year ago, viz, 'The results obtained with Hawaiian soils which are ultra basic in composition must not be accepted as a guide in judging of the state of availability of the elements in old siliceous soils.'" Our provisional method for assimilable phosphoric acid gave more than four times the amount of phosphoric acid obtained by the aspartic acid method, while the amounts of potassium and calcium oxids extracted by the aspartic acid are very similar to those obtained by the weak solvents which have been used by this association.

¹ Jour. Am. Chem. Soc., 22, 285.

² Jour. Am. Chem. Soc., 22, 695.

³ Jour. Am. Chem. Soc., 16, 210.

⁴ A. O. A. C. Proceedings, 1898, p. 63.

REMARKS AND RECOMMENDATIONS.

A study of the work of this association in connection with the determination of assimilable plant food impresses one with the necessity of publishing all of the available data concerning the soils employed which may assist in an interpretation of the analytical results. A simple statement from a referee of the comparative deficiency of a certain ingredient in various soils is not sufficient when this statement may mean the acceptance or rejection of a method which has been tried. The facts upon which the referee bases his conclusions should be either published in his report or reference made to some other published work in which they occur. The amount of assimilable plant food in soils is so liable to fluctuation that even the most complete data obtainable is very apt to prove inadequate.

The wide differences in the results from different chemists working upon the same sample of soil render comparisons of results upon two soils by different analysts practically useless when the content of the soils does not vary widely. The work of the present year would have been much more valuable had the chemists, when undertaking the determination of a given ingredient, not omitted some of the soils upon which the determination was desired. The results of a given chemist working under definite conditions upon a number of soils may be profitably compared with one another, but a glance at the results contained in this report will show that such comparisons were possible in very few cases only. Your referee can do no more than present as a report of progress such data as have been obtained concerning the recommendations received, since the analytical results do not justify drawing any conclusions at this time. The recommendations made to the present referee must remain, therefore, as unfinished business to be carried on by my successor unless he be relieved by the association.

It is recommended that determinations of phosphoric acid be made by N/5 nitric acid as well as by N/5 hydrochloric acid, for reasons which have been mentioned previously.

The recommendation made last year, that a 3-mm sieve be adopted where the soil is to be used for determinations requiring 100 grams or more, is hereby repeated.

The referee's report should contain such data as are necessary for converting the percentage of an ingredient to the number of pounds of the same in a given area of soil at the depth sampled.

At this stage of the proceedings Dr. William McMurtrie, of New York, ascended the platform, at the invitation of the president and amid the hearty applause of the members of the association.

The PRESIDENT. I see the association recognizes an old friend, and it is with very great pleasure that I introduce to the members of the association Dr. McMurtrie, the president of the American Chemical Society.

Dr. McMURTRIE. It is with genuine pleasure, I assure you, that I receive such a greeting from this body to the organization that I have the honor to represent here in an executive capacity. It illustrates very thoroughly to me the interest that you have in the organization of American chemists, and I can well understand the sympathy that you have for it, in that you are surely all members of that organization; if you are not you should be. I can sympathize as warmly with you in the work that you are doing. I was brought up in it, and all of us know how difficult it is to forget the first love.

This discussion that you have just listened to has been particularly interesting to me, because it concerns a kind of work that I had to do in my early experience in connection with agricultural chemistry, and there is nothing, I am sure, that will do as much to advance the agricultural interests of the country. In this statement there is nothing derogatory to the other lines of work which you are doing, and you are to be congratulated upon the opportunities that are afforded you by the enormous success that you have attained in your work in the experiment stations and in the other organizations that you represent. I sympathize fully with you in your work, because I had something to do with the efforts that led up to the establishment of those stations, and I was proud to be associated in that work with such men as Cook, Appleton, Goessmann, and the other men who worked so hard for the passage of the Hatch bill, which brought about the organization of these stations devoted to agricultural science. And we all have a right to be proud of the success that has been attained. We all know how common it was formerly for those wishing further instruction in agriculture to go across the water and seek assistance from our German friends, and surely no one could fail to appreciate and draw inspiration from the work of the leader of agricultural chemistry, the great Liebig. But times have changed and we find that the inspiration and influence are going the other way. We find that our friends across the water come to us for instruction, and we may feel proud that the results of our work have been such that we can confidently point out to them some of the lines in which it will be possible to secure the further advancement of our science.

The changes in the work of chemistry in this country and the great advances that have been made are confined not alone to what has been done in agriculture. It has been interesting to me to follow the work of a young friend who has been abroad for the past two years to complete his education in metallurgical lines. He went to one of the great institutions of Germany, and said that he was greatly disappointed to find that the year's work he was called upon to do was only a repetition of his last year's work in this country. He went to the University of Berlin and attended a course of lectures, and was well satisfied, but when he desired to take up the micrographic study of iron and steel for his doctor's degree and looked around for the material for that work, he found it impossible to obtain the apparatus for the work outside of the Reichsanstalt in Charlottenburg. A few weeks later he met President Low, who said that Columbia University was very well fitted for that kind of work, and he decided to come back to the United States to complete the work for his doctor's degree. It seems to me that these things are always of the greatest encouragement to the chemists of the country.

We have further encouragement, not only in what is being done in research work, but also in applied chemistry. I suppose that in no age of the world, nor in any part of the world, has the progress in industrial chemistry been so great as in this country during the past decade. We find that new industries are springing up all the time; we find that the demand for our products increases at home and increases abroad, and that there is a most gratifying growth in the exports of chemical manufactures. It will interest you, I am sure, to study the statistics of exports of our manufactures of chemical products and note the enormous increase in the last decade. It runs into the millions of dollars, and even into the hundreds of millions of dollars.

Another encouraging thing to me with regard to the growth and progress of chemistry in this country is the demand for expert and accurate work in technology. You are the representatives of probably the most important branch of chemical technology. You know what is necessary to progress; you know what is necessary to the obtaining of good results. You know that all the accuracy of manipulation, all the depths of thought of the careful investigations that belong to any research laboratory, belong to your laboratories; and what you are doing in your laboratories is being done largely in this country in the laboratories of all the leading and successful manufacturers. It is becoming a rare thing nowadays to find an important manufacturing establishment without its research laboratory; and it is interesting to note that what I had occasion to say a few years ago to one of my friends, who was greatly devoted to research work in chemistry and was inclined to sneer at technical work, has been thoroughly borne out. In talking with this young man one day I remarked, "You have had occasion to make some careful determinations of atomic weights; how many determinations do you think it would be necessary for you to make before you would be willing to accept the results?" "Oh, from six to ten," he answered. "Well," I said, "if you were working in my laboratory and couldn't make two determinations and have them agree we wouldn't want you."

The young man's opinion of the work required by the industries has been very materially changed. He has some appreciation of the fact that when a business man asks a question he expects to have that question answered promptly and accurately. It is such questions that come to you, and which you are answering every day, and you may be proud to know that the country feels that you are answering these questions promptly and that you are doing it accurately.

The PRESIDENT. It is impossible for any one man to cover the broad field of chemistry. It is therefore a special pleasure for us to have men in other branches of work, and those who have felt interest in our line of work, look in upon our proceedings and talk to us, and I thank

Dr. McMurtrie in the name of the association for the trouble which he has taken this morning.

Mr. HARTWELL. I have a paper which I would like to present.

The PRESIDENT. We will hear the paper.

A POT EXPERIMENT TO TEST FIELD OBSERVATIONS CONCERNING SOIL DEFICIENCIES.

By BURT L. HARTWELL.¹

Certain impressions which resulted from a study of the results secured by this association over a series of years in connection with soil work were outlined at the San Francisco meeting in July, 1899, in my report² as referee on soil analysis. As the investigation presented in the present paper was undertaken as a result of previous consideration of the points presented at that time, brief mention will be again made of them here. Much analytical work has been done by the association upon samples taken from differently fertilized plots of the same soil. The existence of such plots resulted from a quite extended application of the method of determining the needs of soils by a soil test conducted in the field. Determinations of the total potash, for example, have been made in samples taken from plats which had received no applications of potash and also from those to which potash had been abundantly supplied. In a number of cases the records were sufficiently complete to enable one to calculate approximately the difference to be expected in the percentages of potash in soils from plats receiving potash and from those not receiving it. In making such calculations the applications of potash and the amount of the same removed by the crops during the series of years in which the experiments had been conducted were considered. No data were available for determining the relative loss by drainage, so that the influence of this factor could not be determined. Some of the soils in question were deficient in natural drainage, and it does not seem probable, therefore, that the loss by drainage would be of sufficient importance to explain certain of the differences to be noticed. The figures given below represent the differences obtained by subtracting the percentages of potash found in soil of the no-potash plats from those obtained in soil to which potash had been added. For comparison with these are given also the differences in the net amounts of potash added, found by calculation in the manner referred to above.

Differences between the percentages of potash in the no-potash and potash plats.

Method.	Kentucky soil.	Rhode Island soil.	Indiana soil.	Indiana soil.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Found by J. Lawrence Smith method	.018	— .063	— .081	.078
Found by official method (HCl. 1.115 sp. gr.)	.015	.004	.021	.022
Found by calculation	.043	.046	.008	.018

NOTE.—The results in the case of the official method were deduced from the average of a number of determinations by different chemists. Those by the J. Lawrence Smith method were from the work of single chemists.

¹ H. J. Wheeler rendered valuable assistance by his advice and interest in the work. A. W. Bosworth attended to the drying and weighing of the crops.

² Bul. 57, Div. of Chem., U. S. Dept. of Agr., p. 74.

It may be seen from the above results that there is not the conformity which one would expect between the analytical results and those obtained by calculation. In every case, considering the history of the plats, a plus difference should be expected, as shown by the last row of figures, and yet the total amounts of potash as found by the Smith method are, in two of the four cases, less from the plats receiving potash than from those which did not receive it. The official method does not give the total amount of potash, and the results secured should not be expected to conform to the calculated results as nearly as those by the Smith method. The analytical data here presented do not, as a whole, furnish reliable information as to the excess of potash in one plat over another as a result of increased fertilization with this element. Similar observations were made regarding phosphoric acid. The writer is not inclined to question seriously analytical results representing the average determinations of a number of chemists working with a given sample, although there may be a lack of confidence where the results are those of a single chemist, but in view of the difficulties in obtaining a sample which truly represents a plat of land it seems more probable that in all cases proper care is not exercised in the collection of samples. Failure to appreciate the care necessary in this connection may render valueless the careful work of a large number of chemists.

This association, in its attempts to find solvents which would extract from different soils amounts of potash and phosphoric acid bearing relations similar to the crop-producing power, has compared the analytical results obtained from soils claimed to be deficient in an ingredient with those obtained from soils of high fertility, and consequently containing an abundance of all necessary ingredients. If the solvent failed to extract amounts agreeing with the claimed productiveness of the soils, it was finally abandoned as unsuited for the desired purpose. It becomes evident, therefore, that no doubt should exist as to the amount of the element in question, which is really in a suitable form for the production of plants, otherwise a useful solvent may be discarded because of an incorrect knowledge of the soils upon which it is tried. The soil's natural productiveness as influenced by the particular ingredient under investigation should be known with certainty before any conclusions regarding a solvent or method should be indulged in. The only reliable standard in this work is the crop which a given soil ingredient can produce when the other ingredients of plant food are present in proper conditions and amounts, and when all other factors aside from plant food are favorable. It will be readily seen that such conditions, even though they may be plentiful, are not easily recognized with certainty. It is obvious that a highly productive soil can not be deficient in any necessary element, for the product of a soil as influenced by the plant food contained in the same depends upon the amount of the most deficient ingredient. It does not follow, however, that a less productive soil contains less available plant food, for unfavorable conditions which escape the observer's notice may be the cause of the decreased yield. It must not be concluded that because one soil furnishes 10 bushels of wheat more than another that a similar difference should necessarily appear in the amount of potash or phosphoric acid dissolved by a given solvent. A decidedly unproductive soil may be well supplied with available nitrogen, phosphoric acid, and potash. It is, then, no easy matter to know with certainty the assimilability of a particular soil ingredient, and yet without such assurance it is impossible to judge of the merits of a given solvent for the purpose under discussion. No doubt many incorrect conclusions have been drawn because sufficient attention was not given to finding out the true nature of the soil which was taken as a standard by which to measure the value of a particular solvent.

Perhaps a well-conducted soil test is the most satisfactory method of arriving at a knowledge of the particular deficiencies of a worn-out soil. Trustworthy information can not be obtained, however, in regard to the availability of the nitrogen, phosphoric acid, and potash in a soil under experimentation unless precautions are taken that all other conditions are favorable to plant growth. A test conducted

upon acid soil may, for example, lead to very different conclusions from those which would have been drawn had lime been applied previously in sufficient amount to render the reaction of the soil suitable for the best growth of the crop used in the experiment.¹ There are indications that in some localities magnesia may be lacking in the soil, in which case a soil test should not be conducted without the addition of this constituent.

As soon as the writer received his appointment as referee in the fall of 1898 there was sent to the station chemists a circular letter, published in our last proceedings, which contained a request for soils believed to be deficient in some particular ingredient of plant food. As a result of this inquiry soils were received from Massachusetts and Indiana said to be deficient in assimilable phosphoric acid and two from Connecticut supposed to be lacking assimilable potash. All of the lots were collected in the spring of 1899.

The object in securing these soils was to test them in pots, with special care to supply in the fertilization ingredients other than nitrogen, phosphoric acid, and potash which they might possibly need and which are often overlooked in soil tests. It seemed to the writer, in view of the considerations which have been presented in this paper, that such a course was necessary in order that the assimilability of a given ingredient might be determined under well-controlled conditions and that the samples used for analysis might be taken from the thoroughly mixed soil and leave no doubt about their being representative.

Before describing the pot experiment such records of the soils as it has been possible to secure will be given.

FIELD RECORDS.

*Soil from Wapping, Conn., farm of H. W. Sadd.*²—Sample taken to the depth of 6 inches from seven different points on an acre—a light loam, fine in texture, with a porous subsoil. The field from which this soil was taken, previous to 1891, was an old pasture that had not been plowed or fertilized for years. In 1891 a soil test with indian corn was conducted upon this field under the direction of the Agricultural Experiment Station, Storrs, Conn., resulting in the following yields per acre.³

Yield per acre of indian corn raised in soil test at Wapping, Conn., 1891.

Plat.	Fertilizers supplied.	Shelled corn.	Stover.
		Bushels.	Pounds.
01 } Av.	None	31.1	2,305
A	N	29.6	2,170
B	P ₂ O ₅	30.0	2,130
C	K ₂ O	42.6	2,900
D	N, P ₂ O ₅	37.5	2,680
E	N, K ₂ O	49.4	3,470
F	P ₂ O ₅ , K ₂ O	40.5	3,510
G	N, P ₂ O ₅ , K ₂ O	49.8	4,680
H	Plaster	32.1	2,230

A crop of corn and one of grass had been grown upon the field and an application of stable manure made since the field test was conducted. In taking the sample the manure which then remained on the surface was avoided as much as possible.

¹See Bul. 46, R. I. Agr. Expt. Sta., p. 97.

²Location about lat. 41° 50' and long. 72° 33', on the soil map (Hartford sheet) of the Div. of Soils, U. S. Dept. Agr., and belonging thereby to the Triassic stony loam.

³Storrs Agr. Expt. Sta. Report 1891, p. 192.

Soil from Limerock, Conn., farm of M. H. Dean.—"A striking peculiarity of the soils of this region (the Housatonic Valley) is the presence of large quantities of phosphates. The soil is alluvial, and was apparently formed from the decomposition of limestone formations located along the Housatonic Valley to the northward. This limestone evidently contains phosphates, and by its decomposition these phosphates must have been added to the soil."¹

Soil tests² have been conducted under the direction of the Agricultural Experiment Station, Storrs, Conn., upon the field from which this soil was taken. Below are given the yields per acre:

Yield per acre of maize and rye raised in soil tests at Limerock, Conn., 1889-1893.

Plat.	Fertilizers supplied. <i>a</i>	1889, maize.		1890, maize.		1891.	1892.	1893, maize.	
		Shelled corn.	Stover.	Shelled corn.	Stover.		Un-thrashed winter rye. <i>b</i>	Shelled corn.	Stover.
		<i>Bu.</i>	<i>Lbs.</i>	<i>Bu.</i>	<i>Lbs.</i>		<i>Lbs.</i>	<i>Bu.</i>	<i>Lbs.</i>
0.....	None	9.9	1,470	3.9	1,320	Cowpeas; plowed under; seeded to rye.	3,130	10.3	2,230
A.....	N	16.9	1,890	12.9	1,990		3,740	16.2	2,580
B.....	P ₂ O ₅	9.1	1,670	5.0	1,470		3,840	8.9	2,260
C.....	K ₂ O	18.8	3,340	7.2	4,390		4,400	28.5	4,310
D.....	N, P ₂ O ₅	18.0	2,770	17.0	2,300		4,180	22.0	3,340
00.....	None	14.7	1,790	7.3	1,590		4,060	19.3	2,550
E.....	N, K ₂ O	25.3	2,890	44.4	4,630		4,600	32.6	3,830
F.....	P ₂ O ₅ , K ₂ O	17.4	2,950	8.8	4,300		4,470	36.9	5,250
G.....	N, P ₂ O ₅ , K ₂ O	22.1	3,050	35.0	5,850		4,610	39.5	5,400
Ga.....	N (double amount), P ₂ O ₅ , K ₂ O			40.4	5,860		4,470	42.8	5,800

a The N, P₂O₅, and K₂O were furnished, respectively, by nitrate of soda, dissolved boneblack, and muriate of potash.

b Red clover followed the rye.

Previous to the time of the above tests the soil had been apparently exhausted by cropping without manure. Since 1893 the following crops have been grown without fertilizer, viz: 1894, oats; 1895, rye; 1897, buckwheat, rye; 1898, maize. In 1896 potatoes were grown and one-half ton of a complete fertilizer was applied.

Soil from Amherst, Mass., furnished by the Massachusetts Agricultural Experiment Station from Plat 0, Field F;³ a friable loam, with a retentive subsoil of clay that is nearly filled with gravel and small stones in the lower strata. Previous to 1887 the field had been used as a meadow and was well worn-out, at that time yielding but a scanty crop of English hay.

The plat (No. 5) in proximity to the one from which the sample was taken and with which comparisons will be made later received the following treatment: In 1887 the sod was turned under. The crops grown in 1888 and successively have been Hungarian grass, forage crops, potatoes, wheat, serradella, maize, barley, rye, soy beans, Swedish turnips, and maize. The fertilization began in 1890 with about 500 pounds of dissolved boneblack, 290 pounds of nitrate of soda, and 390 pounds of potash-magnesium sulphate. This application was made yearly till 1895, after which time no more phosphoric acid was added, but the yearly addition of the other ingredients was increased one-half. In 1893 and thereafter the same crops were

¹ Storrs Agr. Expt. Sta. Rept., 1893, p. 123.

² Storrs Agr. Expt. Sta. Rept., 1889, 1890, 1893.

³ Hatch Expt. Station, Mass., 10th An. Report.

grown upon the plat from which the sample of soil was secured (No. 0) as upon the above-mentioned one, but the first addition of fertilizer was made in 1896, since when the application of nitrogen and potash has corresponded with the other plat. So far as is known, no phosphate has ever been added to plat 0. Both plats were limed at the rate of 1 ton of quicklime per acre in the spring of 1898. It may be seen that during 1896, 1897, and 1898 the fertilization of the two plats was the same, but previous to these years plat 0 had received no fertilizer while plat 5, during a series of years, had received a total application of about 1,900 pounds of nitrate of soda per acre, 2,500 pounds of potash-magnesium sulphate, and 2,500 pounds of dissolved boneblack. With the differences in the treatment of the two plats in mind let us compare the yields in pounds per one-tenth acre plat:

Yield in pounds per one-tenth acre plat, Amherst, Mass., soil, 1896-1899.

Plat.	1893, maize.		1894, barley.		1895, rye.	1896, soy bean.		1897, turnips.	1898, maize.	1899, oats.	
	Ear corn.	Stover.	Grain.	Straw.	Entire crop.	Beans.	Straw.	Roots.	Stover, <i>a</i>	Grain.	Straw.
	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.
Plat 0 (N, K ₂ O)	0	366	3.4	28	269	64	182	467	267	107	203
Plat 5 (N, K ₂ O, P ₂ O ₅) . .	349	502	76	217	402	159	318	1,041	457	134	260

a Owing to damage by a violent storm, the stover furnished the best comparison.

I am informed that cabbages were upon the plats in 1900, and that there was almost nothing on plat 0, while upon plat 5 there was a very good crop.

Soil from Orleans, Ind., farm of Frank Turley.—A medium clay resting on dark red clay, which in turn rests on the limestone rock of the region. The combined thickness of the two soils is from 2 to 6 feet. The land has been under cultivation for about seventy-five years, and at one time was so depleted that in an ordinary season it would produce without fertilization only 5 to 8 bushels of wheat. One hundred pounds of ground bone per acre, a quite common application upon this land, increased the productiveness to 15 bushels. A yield of 30 bushels of corn in a favorable season on an unfertilized plat was increased to 38 bushels by an application of acid phosphate and potash. An addition of nitrate of soda gave no further increase, nor did increasing the amount of potash from 30 to 60 pounds per acre. The "Turley" soil upon which some analytical work was done by Huston and Bartlett¹ was taken from the same location as the soil under experiment.

RECORD OF THE POT EXPERIMENT.

As a guide in watering the pots approximate determinations of the per cent of water which the soils were capable of absorbing were made. One hundred parts of the water-free soil were found capable of absorbing the following parts of water, viz: Wapping soil, 63 parts; Limerock soil, 45; Amherst soil, 47; Orleans soil, 46.

All of the soils when tested with blue litmus paper imparted a red color to the same. The Wapping soil, however, gave a more marked change than the others. The humus dissolved by treating a soil directly with ammonia water without previous extraction with hydrochloric acid is sometimes called free humic acid, and is taken as a measure of the need for liming. Qualitative tests with ammonia water were made by noticing the intensity of the color of the extracts. The extract from the Wapping soil was considerably darker than that from the Limerock and Amherst soils. The Orleans soil yielded no color to the ammonia water.

¹ A. O. A. C. Proceedings, 1896, p. 91.

Six Wagner pots, about 8 inches in diameter by 8 inches high, were used for each of the four soils. An amount of the natural soil equivalent to the following weight of completely dried soil was used per pot in each case, viz: Wapping soil, 13 pounds; Limerock soil, 14.2 pounds; Amherst soil, 16.3 pounds; Orleans soil, 13 pounds. In the case of each soil two of the six pots were left without any addition of fertilizer and two received the following:¹

	Grams.
Nitrate of soda	1.0
Dried blood (15.41 per cent N in dry matter)	2.5
Dissolved boneblack	6.0
Muriate of potash	.8
H. G. sulphate of potash	.8
Calcium carbonate, c. p.	10.0
Hydrous magnesium sulphate	1.5

In the case of the Wapping and of the Limerock soils (supposed to be deficient in potash) two of the six pots received all of the above-mentioned materials, except that the potash salts were omitted, and in the case of the Amherst and of the Orleans soils (supposed to be deficient in phosphoric acid) two pots received all of the materials except the dissolved boneblack. This gives, then, in each instance two pots with only the soil as received, two with complete fertilization, and two with complete fertilization except that the supposed deficient ingredient is omitted.

In June, 1899, 50 selected kernels of oats were planted in each pot. When the plants were well up they were thinned to 23 in each pot. Whenever the plants of a particular group required it, sufficient water was added to the pots so that the soil should be about 70 per cent saturated. The weights of the crop at maturity will be given later in this paper in connection with those of 1900.

In the spring of 1900 the soil was emptied from each pot and, after breaking the lumps, replaced. Oats were planted as in the previous year, but the plants were thinned to 35 instead of 23. The only fertilizing ingredient added was 1.5 grams of c. p. ammonium nitrate to each of the 24 pots. This was added in solution at the time of the first watering and was intended to serve as a ready source of nitrogen for the young plants in case the conditions had been unfavorable to the nitric ferment in the dry soil of the pots during the winter season. The crop was harvested at maturity during the first week in August and barley planted in its place. The barley had just begun to head in the most forward pots when it became necessary to harvest it. The weights are not included in this connection because the crop had not had time to dry, and reference to this crop will be made only incidentally when the results bear particularly upon the discussion of those obtained with oats. (See Pl. I, fig. 1.)

Yields (in grams) from the pots containing the Wapping soil.

Materials supplied. (See above.)	Pot No.	1899, oats.				1900, oats. <i>a</i>			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	1	8.25	8.93	3.04	5.89	20.58	23.63	8.63	15.00
	2	7.68	8.28	2.24	6.04	28.39	31.79	11.78	20.01
All <i>b</i> except potash	3	24.00	25.86	9.77	16.09	36.46	41.40	13.01	28.39
	4	23.51	25.37	8.12	17.25	35.32	39.71	11.14	28.57
All <i>b</i>	5	22.31	24.06	7.69	16.37	45.61	51.89	14.34	37.55
	6	22.55	24.39	7.42	16.97	40.73	45.48	16.92	28.56

a All pots received 1½ grams ammonium nitrate.

b Including nitrogen, phosphoric acid, potash, lime, and magnesia.

¹ Multiplying these numbers by 293 gives the number of pounds per acre.



FIG. 1.—OATS GROWN IN WAPPING, CONN., SOIL, 1900.



FIG. 2.—OATS GROWN IN LIMEROCK, CONN., SOIL, 1900.

By reference to the field test with maize on this soil in 1891 (page 75) it may be seen that the results indicated undoubtedly that potash was the ingredient chiefly lacking at that time. The addition of nitrogen with the other ingredients also increased the yield in each case, although to a considerably less extent than the potash did. Phosphoric acid was evidently not needed.

The results of the pot experiment, however, indicate that under the conditions of the experiment no potash was needed the first year for the production of a normal crop of oats when the other ingredients were supplied; in fact, the yield was somewhat lessened where the potash was added. The second year's results, however, showed some gain from the use of potash. In 1899 the yield from the pots (Nos. 1 and 2) containing the natural soil was vastly less than from those receiving everything except potash (Nos. 3 and 4), but in 1900 the relation was much closer. It should be recalled that $1\frac{1}{2}$ grams of ammonium nitrate were added to each pot in 1900; this fact may explain the relative increase upon pots 1 and 2 which had previously received nothing. Considering that nitrogen was somewhat lacking in this soil in 1891, even though sod, which must have supplied this ingredient to some extent, was turned under previous to the field test, it is probable that the lack of available nitrogen in the soil as received was greater than that of potash. It is evident, considering the preliminary tests with litmus paper and with ammonium hydroxid, that the application of some basic ingredient, such as air-slaked lime, is of first importance for the best growth of most crops upon this soil; this is indicated by the fact that the barley, which is more susceptible than oats to acid conditions, failed almost completely in pots 1 and 2.

The only object in view in planning the pot tests with the various soils was to determine whether the supposed greatest deficiency really existed under the conditions of the experiment and to gain some idea of its extent. The number of pots was therefore limited and was insufficient for drawing conclusions regarding other ingredients. The results from three of the four soils have been so surprising that it has been necessary to look for other factors which evidently exerted more influence than those which were expected to control the crop results.

Yields (in grams) from the pots containing the Limerock soil.

Materials supplied. (See page 78.)	Pot No.	1899, oats.				1900, oats. ¹			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	7	12.19	13.23	5.37	7.86	26.78	30.20	11.01	19.19
	8	11.61	12.66	4.92	7.74	25.59	28.61	11.03	17.58
All except potash ²	9	24.89	26.99	9.29	17.70	30.43	34.17	10.59	23.58
	10	24.53	26.49	7.84	18.65	30.88	34.63	11.27	23.36
All ²	11	24.44	26.40	8.54	17.86	31.36	35.35	12.31	23.04
	12	22.77	24.71	7.96	16.75	35.15	39.63	14.14	25.49

¹ All pots received $1\frac{1}{2}$ grams ammonium nitrate.

² Including nitrogen, phosphoric acid, potash, lime, and magnesia.

According to the field tests conducted upon the above soil from 1889 to 1893 (see p. 76), potash was the constituent chiefly deficient. Nitrogen proved of some value, however, even the first year after the original sod had been turned under; the second year its benefit in the production of shelled corn was still more marked, even exceeding that of potash. After cowpeas had been plowed under in 1891 and nitrogen thus supplied, the chief deficiency during the remainder of the experiment was undoubtedly potash. The addition of dissolved boneblack in the earlier years of the experiment seemed detrimental, and there is little evidence at any time that phosphoric acid was deficient. (See Pl. I, fig. 2.)

The above results in the pots are very similar to those with the Wapping soil in showing a slight decrease from the use of potash the first year, but some increase the second year. It may be noticed also that the marked difference in the first year's crop between the yield from the natural soil (pots 7 and 8) and that to which all the ingredients except potash were added (pots 9 and 10) largely disappears after the addition of ammonium nitrate preparatory to the second crop of oats. Considering the results in the field, it seems probable that nitrogen in an available form may have been more deficient than potash when the soil was received. The preliminary tests with blue litmus and ammonia water and the fair growth of barley in the pots to which no calcium carbonate was added indicate much less necessity for liming than in the case of the Wapping soil.

Yields (in grams) from the pots containing the Amherst soil.

Materials supplied. (See page 97.)	Pot No.	1899, oats.				1900, oats. ¹			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	13	14.20	15.27	6.70	8.57	30.11	33.65	14.65	19.00
	14	9.83	10.55	3.94	6.61	36.43	40.83	16.96	23.87
All except phosphoric acid. ² ..	15	27.27	29.62	13.70	15.92	37.21	41.69	17.65	24.04
	16	26.97	29.42	13.22	16.20	39.17	43.35	18.51	24.84
All ²	17	27.43	29.82	11.70	18.12	40.33	44.51	17.11	27.40
	18	24.81	27.12	8.81	18.31	39.32	43.47	16.45	27.02

¹ All pots received 1½ grams ammonium nitrate.

² Including nitrogen, phosphoric acid, potash, lime, and magnesia.

The field results (see page 77) indicate a lack of assimilable phosphoric acid in the plat from which the above soil was obtained, but it may be seen from an inspection of the yields obtained by pot experimentation that no appreciable gain resulted from the application of phosphoric acid (pots 17 and 18) in addition to the other fertilizing ingredients (pots 15 and 16); in fact, the yield of grain during each of the two years was somewhat greater where the phosphoric acid was omitted. The growth of barley was apparently no greater in the phosphoric-acid pots. (See Pl. II, fig. 1.)

The yields of oats in pots 13 and 14 compared with those from the other pots were much greater in 1900 after the addition of the ammonium nitrate than in 1899 when no nitrogen had been applied. It seems probable from this that the soil as received may have been somewhat in need of readily assimilable nitrogen. To be sure nitrate of soda had been applied in the field the spring before the sample was taken, but it is not improbable, considering the readiness with which this compound leaches and its liability to change into less available forms, that an application of readily assimilable nitrogen to pots 13 and 14 in 1899 would have been very beneficial to the crop.

In 1899 oats were also grown upon the field from which the Amherst soil was taken, not, however, until an application of nitrate of soda and potash magnesium sulphate (plat 0) had been made. It is interesting in this connection to compare once more the yield from this plat with that from plat 5, which received dissolved boneblack in addition. The yield per one-tenth acre from plat 0 was 107 pounds of grain and 203 pounds of straw; that from plat 5 was 134 pounds of grain and 260 pounds of straw. Considering the fertilization one might have expected a relation similar to this between the yield from pots 15 and 16 and that from pots 17 and 18, but the above table shows that there was no gain in total crop from the use of phosphoric acid. The application in the pot experiment of calcium carbonate at the rate



FIG. 1.—OATS GROWN IN AMHERST, MASS., SOIL, 1900.



FIG. 2.—OATS GROWN IN ORLEANS, IND., SOIL, 1900.

of about $1\frac{1}{2}$ tons per acre may have increased the assimilability of the phosphoric acid to such an extent that no further application was necessary. To be sure a field application equivalent to 1 ton of quicklime had been made in 18⁶³, but considering the character of the soil it seems probable that the further application in the pots may have changed the reaction of the soil so that the conditions were more favorable than in the field to the liberation of phosphoric acid. The above view is strengthened by the fact that upon certain plats at the Rhode Island Agricultural Experiment Station which had received only nitrogen and potash, and upon which phosphoric acid was lacking, an application of 1 ton of air-slaked lime per acre rendered the phosphoric acid more assimilable but not to the same extent as $2\frac{1}{2}$ tons.

Yields (in grams) from the pots containing the Orleans soil.

Materials supplied. (See page 78.)	Pot No.	1899, oats.				1900, oats. ¹			
		Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).	Entire crop (dried at 100°).	Entire crop (air dried).	Grain (air dried).	Straw and chaff (air dried).
None	{ 19	6.96	7.52	3.31	4.21	10.57	12.15	5.09	7.06
	{ 20	6.53	7.11	2.86	4.25	11.11	12.72	4.93	7.79
All ² except phosphoric acid..	{ 21	8.05	8.74	4.22	4.52	10.97	12.39	4.88	7.51
	{ 22	9.02	9.86	4.04	5.82	9.24	10.55	4.44	6.11
All ²	{ 23	23.38	25.31	9.56	15.75	28.82	32.44	14.18	18.26
	{ 24	24.19	26.25	10.26	15.99	28.88	32.24	13.33	18.91

¹All pots received $1\frac{1}{2}$ grams ammonium nitrate.

²Including nitrogen, phosphoric acid, potash, lime, and magnesia.

From the field observations upon the soil heretofore given it was found that nitrate of soda gave no increase, but that an application of phosphoric acid resulted in a greatly increased yield.

The above results in the pots confirm without doubt the field records concerning the lack of assimilable phosphoric acid, for the entire crop from the pots (Nos. 23 and 24) receiving all the ingredients was nearly three times as great as from those which were treated the same, except that phosphoric acid was omitted (pots 21 and 22). The lack of phosphoric acid is so marked in this soil that it seems to be about the only addition necessary for the production of a maximum crop of oats. (See Pl. II, fig. 2.)

The Indiana soil is then the only one of the four upon which the results of the two years' experiment in the pots with oats agreed fully with the field observations. Many factors may have had an influence in causing the apparent disagreement between the field and pot experiments. In the pot experiment the soil was thoroughly mixed and all large lumps broken; water was supplied whenever needed by the plants; temperature conditions were different in the exposed pots than in the earth; the crop was different in some cases from those used in the field. It can not be denied that these factors may have exerted an influence, but in view of the fact that soil tests conducted in pots have in so many instances borne out the conclusions derived by field experiments similarly conducted at the same time it seems more likely that most attention should be given to the following considerations, viz: Lapse of time since the field tests were conducted and the addition to certain pots of all ingredients which might prove beneficial other than the one supposed to be deficient. The field test upon the Wapping soil was conducted only one year and as long ago as 1891 and the last year of the field experiment upon the Limerock soil was 1893. The chief deficiency formerly noticeable upon these soils was potash, but nitrogen was needed to some extent. It is reasonable to suppose that recent cropping and man-

agement may have changed the conditions to such an extent that readily assimilable nitrogen, as was indicated by the pot experiment, is at present more deficient than potash.

The preliminary tests to ascertain the need of alkaline treatment led to the addition of calcium carbonate to the pots from which conclusions were to be drawn as to whether the supposed deficiencies existed. Other ingredients sometimes omitted in field experiments were also supplied. These additions were no doubt somewhat responsible for the exceptional results obtained in the pot experiment. Before experiments are conducted to ascertain food requirements of soils all other conditions should be made as favorable as economy will permit. It is obviously irrational to purchase plant food which may prove unnecessary after mechanical conditions, drainage, and the reaction of the soil are made favorable.

The referee on soil analysis has usually been expected to place samples of soil in the hands of the cooperating chemist a few months after receiving his appointment. It is obviously impossible under such circumstances to do more than canvass for soils upon which field experiments have been conducted, and which are thought, from the records at hand, to show a deficiency of some particular ingredient. Time scarcely remains for the referee to make a complete study of the detached field records, to say nothing of conducting experiments under his own supervision, which shall be especially designed to answer the particular question to be decided.

It seems probable, in view of the data presented in this paper, that much of the chemical work of this association has been done upon samples of soils which did not possess the characteristics attributed to them, and that hasty conclusions have therefore been drawn regarding the efficiency of certain methods. It seems too much to expect a method to answer questions regarding the assimilable nitrogen, phosphoric acid, or potash in a soil which needs draining, liming, or the addition of other ingredients aside from the so-called essential elements to put it in the best condition for the growth of most agricultural plants. Every crop the quantity of which is to be taken as a standard in judging the value of a general method for determining the particular requirements of a soil for assimilable nitrogen, phosphoric acid, or potash should have been grown after all general conditions of the soil had been made favorable for the full development of the plants.

SUMMARY.

The analytical results secured by this association have frequently failed to account satisfactorily in the case of given soils for differences caused by known fertilization and cropping.

This failure was probably due in many cases to the fact that the small amount of soil analyzed did not properly represent the large area from which it was taken.

Land upon which field experiments are to be conducted for showing its need of plant food should first receive such treatment, other than the application of the particular ingredient to be studied, as is economical and necessary for the proper growth of most agricultural plants.

Soils designed for use in testing methods for determining assimilable plant food should in many cases be subjected to pot experimentation before they are distributed by the referee.

Mr. PATRICK. In view of the fact, as brought out in the paper just read, that a number of years elapsed between the liming of the fields and the taking of the samples, cropping had occurred right along, and perhaps some barnyard manure applied, it does not seem to me at all strange that the pot results did not agree with the field results. This paper enforces very strongly the teaching that the soils for experi-

mentation should be taken from the field immediately after the crops are taken and the crop report is made from the field.

MR. WHEELER. I think attention ought to be called to the fact that in the Limerock soil, which is supposed to be deficient in potash, no potash was subsequently applied and crops were grown continuously which would take out more potash. Notwithstanding these facts, we find that additional potash is not needed. It seems to me that the results point out the fact that we must be very careful about conducting our soil tests and know absolutely the deficiencies of the soil before we use it as a basis for comparing the merits of our analytical methods.

MR. PATRICK. What the last speaker has said about the potash may be true; nevertheless, we observe in the first two soils that nitrogen was the thing that was needed. It was the presence of ammonium nitrate that brought out the fertility of that soil and not the addition of potash. Furthermore, we have always been taught to believe that soils develop available plant food in the course of years, especially when crops are grown upon them and the roots permeate the soil to a considerable extent, the roots not being easily removed with the crop.

MR. HARTWELL. In order to bring the point once more before the association, let me say there was no attempt made to create a conflict between the pot and field experiments. We simply stated what the field experiments indicated at such a time and under certain conditions. The same crop was not tried in the pot experiments as was tried in the field experiments. The principal point is that we can not accept field results which were conducted some years previous as an indication of a lack of a particular ingredient in the soil at the time the sample is taken and distributed. Of course, it is well understood that one crop may cause a gradual deterioration in one ingredient of the soil and another may exhaust a different constituent.

MR. SHUTT. I think one of the most important features of the referee's report is a certain suggestion which he made with regard to the statement of results of available plant food. He favors the idea of having them stated in pounds per acre. I think it a very wise suggestion to give the depth of the surface soil and also the weight of a cubic foot of the soil in stating these results. It would be unwise to adopt the method of stating the results as pounds per acre and reject that of the percentage of water-free soil, because the water content of a soil varies from day to day; it may be from 20 to 40 per cent of water would have to be taken into consideration when the weight of a cubic foot of soil was taken and the percentage of moisture would have to be calculated. The same is true with regard to the depth of the soil. We should have to reduce them all to the same depth as well as weight.

It seems wise to retain the plan we have now for stating the results on the water-free soil, and at the same time to accept the suggestion

of making a statement in regard to the depth of the surface soil under consideration and its weight.

There is another question. The root systems of different plants have different depths, and consequently plant food available to one crop would be out of reach of another crop. It does not hold good in every case that we should state the amount of plant food available per acre. It seems to me that none of the results, as stated by the referee in this table, would justify us in excluding the citric acid method from consideration. Unfortunately, very few of the results have been carried through by the same analyst. There is really nothing which would prevent us from accepting the suggestion to state availability of plant food in the soil; nothing to prevent our accepting the citric acid method as being as accurate as the nitric acid, or fifth normal hydrochloric acid method. I certainly think that we have in a measure lost sight of what we started with in the estimation of available plant food. We have considered the soil instead of the plant. You will remember this whole affair started with the determination of the acidity of the plant-root sap. We supposed the plants received their nourishment from the soil through osmosis, consequently it seems to me the legitimate way would be to ascertain the foraging power of a plant in that connection. It is a matter of regret to me that the results published by Dr. Dyer in 1894, and which gave us the incentive, have not been corroborated or confirmed or duplicated on this side of the water. I have been using the citric acid method because there is really a scientific basis for it. I have used it continuously, and have never seen any scientific basis for the adoption of the ammonium chlorid, calcium chlorid, and other methods which have been introduced in the past few years. I should be sorry to see the citric acid method dropped. There is nothing to warrant such drastic measures.

MR. HARTWELL. I think the last speaker has failed to understand my position in regard to the question. I simply stated that the abandonment of the citric acid method had been recommended by former referees, and I also said that the results at present were not conclusive. I, for one, do not believe in dropping any of our methods which do not present any analytical difficulties, until we are very sure about the nature of our soil.

MR. DYER. I would like to claim your indulgence to say a few words on this subject; one of the greatest interest to me. I would like first to point out that in this series of analyses the 2 per cent citric acid solution has been used. That is not at all the same as the original 1 per cent citric acid method as proposed by myself. I should like to take this opportunity to make an explanation about that original process, and in regard to the way in which it has been applied by your association in some of the cooperative trials that have been made. My process was originally, and still is, the use of a 1 per cent citric acid solution, a

definite volume being used for a definite quantity of soil, without relation to basicity of the soil in the matter of carbonate of lime. When I read my paper at the chemical society what seemed to me a very pertinent suggestion was made. It was that, as carbonate of lime neutralized citric acid, a sufficient quantity of acid should be used to neutralize the carbonate of lime in the soil and then enough citric acid should be present to leave the solution acid to the extent of 1 per cent. A great deal of work has been done since then by Dr. Woods, of Cambridge, on various soils in India using this process, and there were certain soils remarkably poor in phosphoric acid and some were undoubtedly poor in potash, as tested by field results, and in comparing these soils with other soils by the citric process these differences were not brought out as they should have been. He had used the modification which allowed for neutralization of the carbonate of lime. Those soils happened to be soils containing a good deal of chalk. It is the process as originally suggested in my paper, without allowing anything for carbonate of lime, which gave results according so beautifully with the field results that we might infer one from the other. He made what seemed a very pertinent suggestion. It was that solvents being the result of an attempt to do something that accorded with the sap acidity, it was right surely that the carbonate of lime should not be corrected. What I mean is this: If the plant gets its mineral food by means of the acidity of its sap, then an excessive quantity of carbonate of lime in the soil is a natural difficulty with which the plant has to cope. If we wipe out this difficulty by neutralizing the carbonate of lime, we are departing from the original principles of the process. Again, if we allow for the carbonate of lime we get a much larger quantity of citrate in the solution. Of course, whatever process is used must be an arbitrary one. The love of offspring is at once a virtue and an infirmity in that it is so strongly planted in the human breast. We never like to think that other peoples' children are better than our own until we have overwhelming evidence, and then we are obliged to give in.

I have a message to deliver to you, and a suggestion to make. Very shortly before his death Sir John Lawes, and also Sir Henry Gilbert, urged upon me strongly that if good fortune should bring me into contact with you in your annual convention I should make a strong recommendation on the subject of soil sampling. That is, as you have done so much work in the direction of bringing about uniformity of analyses, to see whether it is not possible for you to do something to bring about uniformity of soil sampling. In connection with some of the questions that have cropped up to-day my message comes in pertinently. I am afraid to say how many thousands of analyses we have made of certain soils. Almost every difficulty connected with soil sampling has necessarily been considered. In the earlier years of

sampling at Rothamsted samples were taken of the surface soils without reference to the fact that the surface soil differs considerably in composition with each inch of depth. It is for this reason necessary that the sample should be taken in the shape of a cube or cylinder, all the cross sections of which shall have an equal area. Only when the sample is of this shape will the successive layers of soil be taken in their due proportion. Although attention has been very frequently directed to this matter, there is too much reason to suppose that not a little otherwise good soil work has been vitiated by reason of insufficient attention to the drawing of representative samples.

In 1856 the method of collection then and subsequently adopted was devised, and I, perhaps, can not do better than quote directly from Professor Warington's lecture (U. S. Department of Agriculture, Experiment Station Bulletin No. 8, 1892, p. 39). Professor Warington says:

A frame made of stout sheet iron, in shape a rectangular prism, open at top and bottom, is driven into the soil by repeated blows of a wooden rammer till the soil has the same level inside and outside the frame. The soil inside the frame is then cut out and constitutes the sample of the first depth, or surface soil. That the frame is accurately emptied is ascertained by trials with a wooden gauge of the same depth as the iron frame. If a sample of the next depth is to be taken the soil is cleared away around the outside of the frame till the level is reduced to that of the bottom of the frame; the frame is then driven down again and the former operations are repeated.

Soil sampling at Rothamsted is usually carried down to three depths, but in a good many cases it has been carried down to twelve depths. The area of the sampling frame used for the first depth is usually 144 square inches (12 by 12 inches), a smaller frame (6 by 6 inches) being used for the succeeding depths. * * *

The iron frame has a stout rim along its upper edge to increase its strength. The best sampling frame is made of cast steel; this form of frame needs no rim. (Models of both the larger and smaller steel frames used at Rothamsted were on this occasion presented to the U. S. Department of Agriculture.)

When the soil sampling is carried below the first depth care must be taken when digging around the frame that each depth of soil removed is placed by itself, so that when the pit is filled in the soil may be returned to its proper position. A record must be kept of the place where the sampling was conducted, as a soil can not be accurately sampled twice in the same place.

Each sample of soil is weighed as soon as it is removed from the frame, and is put into a bag by itself. When the soil reaches the laboratory it is at once broken up by hand, into small pieces, and laid on paper trays, which are placed on the shelves of a storeroom kept at a temperature of about 55° C. till thoroughly dry; each sample is then returned to its bag. This immediate drying of the soil at a low temperature is essential if changes in the organic matter, and especially nitrification, are to be stopped. This practice dates at Rothamsted from 1877. After drying the soil it may be stored till leisure is found for further work. Each bag is then weighed. The soil is crushed and passed through a $\frac{1}{4}$ -inch sieve; the stones that do not pass through this sieve are weighed (and subsequently described) as stones. All that passes through the sieve is thoroughly mixed and a sufficient quantity is finely powdered for analysis. Mixed samples are prepared after the soil has passed through the $\frac{1}{4}$ -inch sieve or after it has reached the stage of fine powder.

This method of taking soil samples, with the exception of the adoption of the definite temperature above mentioned for drying, has been, as has already been said, in use at Rothamsted since 1856, and it is interesting here to record that between 4,000 and 5,000 individual samples of the Rothamsted soils have been collected in the fashion above described.

The depth of 9 inches fixed for sampling the surface soil is somewhat greater than would now be adopted in the light of the experience that has been gained, although it would now be obviously inexpedient to make any change at Rothamsted, since in that case future samples would not be comparable with earlier ones. But in new soil work begun elsewhere both Sir John Lawes and Sir Henry Gilbert came to the conclusion that about 8 inches would be a fairer depth at which to mark off the surface soil from the subsoil. And since the metric system is so widely used in scientific work, the recommendation made by them to those initiating experimental soil work is to take the nearest round-number metric approximation and adopt a cube with the linear dimension of 0.2 meter as the unit for soil sampling. This would give to each layer of soil a depth of 7.874 inches, or practically 8 inches.

Of course, I can not make a recommendation, but I can ask you, if you think favorably of what I have said, to request some one to make a motion referring this question of uniformity of soil sampling, as well as methods of analysis, to a committee who should be empowered to thoroughly go into the question.

Mr. MACFARLANE. I would like to express my thanks to Mr. Hartwell for having brought forward such a vast amount of information for us to digest. Generally speaking, his results seem to confirm a great deal of Wagner's teaching. We all know that Wagner has given us much information in regard to the application of fertilizers to soils, and has especially laid great weight upon the necessity for first attending to the physical and other conditions of the soil before we can expect that the consideration of its chemical constituents and the application of fertilizers will produce any great results. This point has been brought out by Mr. Hartwell in the pot experiments. It seems to me they settle in a great degree the question of plot versus pot in favor of the pot experiments. This belief was strengthened by my visit to Darmstadt during the past summer, where I saw Professor Wagner's pot experiments. I was very much impressed by these experiments, and the influence which a very slight addition of a fertilizing constituent has on the growing plant. There was one particular instance of the addition of a small amount of phosphoric acid to a certain pot which produced a very appreciable difference in the condition of the plant, and yet it would have been altogether impossible to have detected by analysis any additional phosphoric acid in the soil which was operated upon. That ought to be considered

in studying the facts presented by Mr. Hartwell. I can only say that I have profited very much by the facts and figures which Mr. Hartwell has brought forward.

MR. EWELL. I move that the referee on soils for next year be instructed to consider the adoption of methods for the mechanical analysis of soils and for the statement of the results thereof. I should like to enlarge that motion to include the method of sampling soil as suggested by Mr. Dyer.

The motion was referred to the committee on recommendations.

The report on liquor and food adulteration was then called for, but no report was presented, the referee not being present.

MR. WINTON. Before we leave this subject, which has been so thoroughly treated, I would like to say a word with reference to a division of the work on food adulteration. Anyone who has done work in this line appreciates the vast number of methods that must be employed. I have had some experience in the analysis of fertilizers and food products, and I think I can say, without exaggeration, that in the examination of food products with reference to adulteration the analyst is called upon to employ ten times as many analytical processes as in the examination of fertilizers. In addition to that, many of the methods which he uses are very little understood. There is a vast amount of work before us if we are to get methods in shape for the examination of food products. It is unfortunate that there is no referee at this convention on this important subject. The amount of work to be done is so large that it is a great pity we must lose the work of this entire year. To return to the suggestion that the work be divided—we might make a division of this work and the president be empowered to appoint a number of referees. That would accomplish the result, perhaps, but I would suggest that a better plan seems to be that we have but one referee, but that that referee be instructed to divide the work and to allot different sections to different men. By this means the whole field could be covered, and yet in a uniform way. These different subreferees could meet together under the general guidance of the referee and accomplish in a year a large amount of work. I will therefore introduce this resolution:

Resolved, That the subject of liquor and food adulteration be subdivided as follows: (1) meat and fish; (2) fats and oils; (3) cereal products; (4) infants' and invalids' foods; (5) saccharine products, including confectionery; (6) canned vegetables; (7) tea, coffee, cocoa, and the like; (8) spices and condiments; (9) vinegar; (10) flavoring extracts; (11) fruit products; (12) fermented and distilled liquors; (13) baking powder and baking chemicals; (14) preservatives; (15) dyes. That it be deemed the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate a certain subject or subjects and prepare a report.

You will notice that the subject is divided into 15 sections, and perhaps you are all aware that there are only four or five food analysts

in the association at the present time. From this it is evident that each one of these food analysts may get an appointment. The idea is that, although the number of workers is limited, the work can be satisfactorily carried on by giving each one a number of subjects; or, if a subject does not require especial investigation during a particular year, it can be kept out of sight entirely. I therefore submit this resolution.

Resolution seconded.

Mr. PATRICK. I wish to ask in what section, in that list of fourteen or fifteen subjects, do you place such articles as butter and cheese?

The PRESIDENT. May I suggest that the whole question be referred to a special committee to be reported upon at the afternoon session?

Mr. WILEY. I would like to suggest the advisability of including in that committee the referee for dairy products, so as to bring the whole matter under one head.

Mr. LEECH. That work is so extensive in itself it would seem worthy of having a separate referee. I agree with Mr. Winton that the entire food work is so broad that it should be subdivided. It is impossible for one person to cover the ground.

Mr. PATRICK. If I heard correctly, distilled liquors, wines, etc., were included. That is a rather large subject and one on which we already have a referee.

Mr. WINTON. I would state the reason for not including dairy products under this head. We examine dairy products with two objects in view: First, the detection of adulteration, and second, the determination of the nutrition contained in the milk. That is, it is not a question of adulteration alone, and for that reason only a part of the work would come under this head, and this was thought of in making the classification.

The resolution was referred to a special committee.

The PRESIDENT. We have already had the pleasure of a visit from the president of the American Chemical Society, and I see before me this morning the past president of the Association of American Agricultural Colleges and Experiment Stations. I am sure the association will be pleased to hear from Mr. Stubbs.

Mr. STUBBS. Mr. Chairman and gentlemen of this association: I am in mind of the time when I began to learn to swim. I had a fine teacher, consequently one without any heart, and when he threw me into the water he said: "There, swim!" Now, in the presence of this body of scientific men, whose work I appreciate as never before, whose influence upon the common welfare of society is already so great and yet destined to be so much greater, I feel that the platform is no place for me, but that I should be in the body of the audience listening. I am only sorry that duties pertaining to the work at New Haven made it impossible for me to get here in time for the work of this associa-

tion beginning yesterday. I made all possible haste to get here in order that the Agricultural Experiment Station of Nevada might be represented, not worthily, but still represented.

I am very glad indeed that I belong to the ex's. I am reminded of a little story that is told of Senator Hoar. One of the ex-Senators called upon him at the Capitol, and having, of course, the privileges of the floor, went into the Senate Chamber and visited with him. As they were about to retire, the ex-Senator politely stepped back, saying: "After you, wise Senator." The grand old man from Massachusetts straightened up and replied: "Oh, no! the x's always go before the y's." Therefore, I am glad to meet you.

The PRESIDENT. We still have a few minutes before adjournment, and I will call for the report of the committee on recommendations.

Mr. WILEY. After we adjourned last evening the board of managers of the Cosmos Club extended the courtesies of the club to the members of this association during their stay in the city. We shall be glad to have you visit the club, where you will find papers, magazines, writing paper, etc., and something to eat and drink also, according to your taste, and where I hope you will be comfortable and where I know you will be welcome.

The report of the committee on recommendations was then read by the chairman, Mr. Huston.

REPORT OF THE COMMITTEE ON RECOMMENDATIONS.

NITROGEN.

(1) It is recommended that the neutral permanganate method (Street) be referred to the referee for 1901.

Adopted.

(2) It is recommended that the alkaline permanganate method (Jones) be referred to the referee for 1901.

Adopted.

PHOSPHORIC ACID.

1. On page 13, Bulletin No. 46, Division of Chemistry, U. S. Department of Agriculture, under the heading (b) Optional Volumetric Method. (1) Preparation of reagents, omit the following:

(b) Potassium nitrate or ammonium nitrate solution for washing. Dissolve 3 grams of the salt in 100 cc of water.

(c) Nitric acid solution for washing. Dilute 100 cc of nitric acid (specific gravity, 1.42) to 1 liter with water.

Adopted.

2. On page 14, Bulletin 46, under the directions for the determination of phosphoric acid (P_2O_5) by the optional volumetric method, after paragraph b, add: "b₂. Proceed as directed in b₁ with this exception, heat in water bath at 45° to 50°, add the molybdic solution, and allow to remain in the bath, with occasional stirring, for thirty minutes."

Adopted.

3. Following paragraph b_2 add: " b_3 . Proceed as in b_1 to the point where the solution is ready to be placed in the water bath. Then cool solution to room temperature, add molybdate solution at the rate of 75 cc for each decigram of phosphoric acid present, place flask containing solution in shaking apparatus and shake for thirty minutes at room temperature, filter at once, wash and titrate as in preceding method."

Adopted.

Mr. HUSTON. The recommendation of Mr. Macfarlane would entirely change our methods for the determination of phosphoric acid, and that, of course, could not be done under the constitution without at least one year elapsing.

Mr. Wheeler's resolution is that we introduce a method for the examination of basic slag and make it a provisional method. The committee wishes to report that it recommends that the association instruct the referee to investigate the methods for the determination of availability in basic slag. It does not think it advisable at this time to adopt a provisional method.

Mr. BARTLETT. We shall probably have some goods of that nature to analyze, and would it not be well to authorize us to use some method, perhaps that of simply determining the total phosphoric acid, as we do with bone meals?

Mr. WHEELER. I will state my reason for introducing that motion. Basic slag is now being shipped into California. It is also being shipped into the Southern States and is likely to be shipped into other States. It seems that in the interim which will elapse before we can investigate and adopt some method as official we might adopt the same method which prevails in Germany, so we can examine these goods by the same method they will be tested by before shipment here. In view of the fact that the 2 per cent citric-acid solution gives comparative results, I think we could safely adopt it if we so desire, not as an official method, but simply as provisional, until we can adopt something to which we can give our approval.

Mr. MACFARLANE. I would like to say that I consider the recommendation of the committee eminently wise, and I hope Mr. Wheeler will not insist upon the adoption of a provisional method. The analyst should rest content with determining the total phosphoric acid, and let basic slag stand on its own merits.

Mr. HUSTON. I will say that we spent quite a long time considering the matter, and had many reasons for making this recommendation. In the first place, if you are to attach the same commercial value to available phosphoric acid in basic slags that you do to available phosphoric acid in other goods, the 2 per cent citric-acid solution gives a value to basic slag out of proportion either to the cost of production or its value in the field. I believe if you will thoroughly leach out basic slag with water you can remove a large portion of the free lime. With a more careful treatment with water you will reach a result on

citrate soluble phosphoric acid nearer the commercial value and field value than by treating with a 2 per cent citric-acid solution. I believe it dangerous to adopt it as a provisional method at present. Pressure will be brought to bear to retain it as official. We ought to give the matter immediate and serious attention.

I think we would probably have to use more water and grind the material with the water, as in the method with acid phosphate. I believe a little manipulation of that sort will remove more of the free lime. Work in our laboratory shows that it takes out a large quantity.

There is another point in connection with availability, and that is the product of the reaction of neutral ammonium citrate on basic slag is not very soluble in the fluid itself, and where you take 100 cc of our 0.109 solution and treat 2 grams of slag with it you get a certain percentage soluble in that reagent. If you will add 400 cc of water, you will get a very much larger solubility, which convinces me that the product of the reaction is not soluble in the more concentrated fluid.

MR. MACFARLANE. A word of caution in regard to the use of water in determining the available phosphoric acid. We have tried it and find it to be impracticable. You might, by moderate washing, say of several hours, increase the available phosphoric acid 1 or 2 per cent, but you can not by that means remove the free lime to the necessary extent. I could not recommend the chemists present to waste their time trying to remove all the free lime.

MR. SHUTT. In this matter the agricultural chemist stands between the consumer and the vender. This basic slag is an imported article here, as well as with us. The universal method of analysis in Europe is the citric-acid method. When the goods come to be tested on this side we shall have to explain why our results do not coincide with those obtained by European chemists. The method seems to have some things in its favor.

MR. HUSTON. If these goods come to the State of New Jersey, they will have to be treated at 40°. Our American manufacturers know what to expect when they ship goods to New Jersey. The foreign manufacturers will speedily become acquainted with the methods to be used on the basic slag, and will conform to our methods, or, at least, will know what to expect when the materials get here. I believe the citric-acid method gives results too high.

MR. DYER. Are we not sometimes in danger of confusing two things, viz, availability and value? Phosphoric acid in two forms might be equally available ultimately, but the value might be different. It is well recognized, from the experiments of Wagner as well as other experimenters, that what we call the available phosphoric acid in slag has not the value to the farmer of a like quantity of phosphoric acid in the form of superphosphate. It might be equally available when the plant can get it. Surely the question of the value of a unit is a different question from availability.

Mr. MYERS. It seems to me that this difficulty can be overcome by the State chemists, until they are ready to adopt some fixed policy, by treating basic slag about as they would floats or finely ground bone meal. It is a special product and has to be treated by a special method, and as a basis of valuation you can take the total phosphoric acid, and if the manufacturers want to claim more, then that is their misfortune or their good luck, as the case may be. At the same time the farmer knows that he is receiving a total quantity, which upon certain classes of land becomes rapidly available. There is no fraud practiced on anybody, and by changing the scale of valuation, for a valuation has to be fixed, as suggested by Mr. Dyer, the matter can be bridged over until such time as the association can establish a satisfactory method of analysis. I agree heartily with Mr. Huston that the method of analysis as adopted in Europe is not a satisfactory method. I think that the acid solution shows too much phosphoric acid. I do not think that is the method which will finally be adopted by this association, but I know of no method that can be offered now except, as Mr. Wheeler says, provisionally, and it is a question whether a provisional method, that may cause trouble later, ought to be put on the books. Anybody can determine the total phosphoric acid and report on that just as we do on total phosphoric acid in floats and in fine bone meal. It would be well also to note in the analysis the proportion of finely ground meal in the Thomas phosphate, because the availability of Thomas phosphate is undoubtedly affected very largely by the fineness of the powder in which it is presented to the farmer.

Mr. WHEELER. I think the members of the association are aware that there is a great difference in the assimilability of the phosphoric acid in basic slag. The slags, which contain a considerable amount of silica, are much more assimilable than those where a less quantity of silica is present. I would like to ask the gentlemen of the committee whether they can tell us positively that with two classes of slag the one containing less silica may be finer than the other and still be less assimilable? It seems to me the answer to that question should be a positive one.

Mr. HUSTON. We have no American data on the subject.

At this point the meeting was adjourned to meet at 2 o'clock.

SATURDAY—AFTERNOON SESSION.

The meeting was called to order by the president at 2 o'clock.
The PRESIDENT. We will now have the report on ash.

REPORT ON ASH.

By A. E. SHUTTLEWORTH, *Referee*.

The following circular letter and outline of the work proposed were sent to a number of American chemists most likely to cooperate with us:

MY DEAR SIR: At its recent meeting in San Francisco, the Association of Official Agricultural Chemists, considering the subjects of soil and ash analysis of sufficient importance, provided for the appointment of separate referees.

Dr. G. S. Fraps, of the North Carolina College of Agriculture, appointed associate referee of ash analysis, and myself, appointed referee, have pleasure in inviting you to assist us in this important work by conducting one or more of the investigations as outlined on inclosed sheet.

Upon the receipt of your reply informing me that you will join in this work, and indicating the investigations that you have chosen, I will immediately express to your address the necessary samples and forms.

Yours, very truly,

A. E. SHUTTLEWORTH.

PROPOSED RESEARCH WORK.

1. (a) Make by the official method (see "Methods of Analysis," 1899, p. 77) at least three crude ash determinations in sample No. 1, using for each 12, or approximately 12, grams of the material.
- (b) Determine the carbon and the carbonic acid in this crude ash, reporting on Form I, per cent of crude ash, per cent of carbon and carbonic acid in crude ash, and per cent of carbon-free ash, which is the crude ash less the carbon and carbonic acid.
2. (a) In each separate ash prepared under 1, determine the total silica (i. e., make no separation of crude silica into soluble and insoluble), reporting on Form II crude silica in carbon-free ash.
- (b) In sample marked No. 2 make three total silica determinations similar to (a) under 2, reporting results on Form II.
3. (a) Determine by the official method the per cent of K_2O in each sample of ash prepared under 1, reporting on Form III per cent of K_2O in carbon-free ash.
- (b) In sample 2 make three K_2O determinations similar to (a) under 3, reporting results on Form III.

Notes.—1. In each determination, (a) and (b) of 2, not less than 1 gram of the ash is to be used, and the filtrates from these may be employed respectively for (a) and (b) under 3.

2. To determine carbonic acid see 6 on p. 79 of "Methods of Analysis," 1898.

3. In (a) and (b) of 2 collect in a tared Gooch crucible and dry to constant weight at $110^{\circ}C$. After incineration the loss in weight gives the carbon.

4. Results are requested on or before July 1.

REMARKS.

Eight chemists replied that they would undertake the work, and asked that samples be forwarded to them. The samples were forwarded on March 30, together with the following letter of explanation and blank forms for reporting results:

MARCH 30, 1900.

DEAR SIR: In response to your letter of recent date, I am sending to you by to-day's express samples marked No. 1 and No. 2 for A. O. A. C. work, as outlined in my circular letter already received by you.

In calculating your results, please observe that sample No. 1, which is prepared from oat straw, contains of water-free—that is, of dry substance—95.15 per cent.

Sample No. 2, which is ash prepared from the same oat straw by the use of calcium acetate solution in my platinum ash apparatus (see pp. 304 and 305 Experi-

ment Station Record, Vol. XI, No. 4), contains 86.61 per cent carbon-free ash. Please calculate results on the basis of 86.61 per cent carbon-free ash. The remaining 13.39 per cent are composed of the following:

	Per cent.
CaO from added calcium acetate solution.....	2.50
CO ₂ from added calcium acetate solution.....	10.15
Carbon from added calcium acetate solution.....	.74

We hope to ascertain by these tests whether the ash which you prepare from sample No. 1 by the official method contains the same percentage of SiO₂ in the carbon-free ash as No. 2. These tests are to see whether ash prepared by the official method is quite decomposed by hydrochloric acid or not. If it is not, then SiO₂ determinations are too high, owing probably to chemical combination of SiO₂ and bases during incineration.

We hope further to ascertain whether part of the potash is either volatilized or united chemically with SiO₂ by incinerating to an ash.

Inclosed please find forms upon which please report your result.

I am, very respectfully yours,

A. E. SHUTTLEWORTH.

RESULTS REPORTED.

Owing chiefly, I suppose, to pressure of work, only three of those to whom samples were sent reported results. These are Dr. G. S. Fraps, assistant chemist North Carolina College of Agriculture and Mechanic Arts, Raleigh, N. C.; Mr. C. C. Moore, of the U. S. Department of Agriculture, Division of Chemistry, Washington, and William P. Gamble, B. S. A., special assistant in the department of chemistry, Ontario Agricultural College, Guelph, Canada. To these gentlemen we are indebted for the valuable results contained in the following tables:

TABLE I.—Carbon-free ash in sample No. 1.

Analyst.	Determinations.	Percentage in water-free substance.			
		Official method.		New method.	
		Single determination.	Average.	Single determination.	Average.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moore, C. C.	1	6.25	} 6.25		
	2	6.25			
Fraps, G. S.	1	5.56	} 5.55		
	2	5.59			
	3	5.49			
Gamble, Wm. P.	1	6.40	} 5.87		
	2	5.01			
	3	6.20			
	4	5.66	} 5.655		
	5	5.65			
	6			6.38	} 16.38
	7			6.39	
	8			6.36	

¹ Prepared from sample No. 1 by the addition of calcium acetate in a closed platinum apparatus.

Crude ash of vegetable and animal substances, ever so carefully burned, contains varying quantities of carbonic acid, carbon, and foreign matters. Therefore percentages determined and stated as crude ash may vary as much as 0.2 to 2 per cent in the same substance. Because of this I think chemists should at least make carbonic acid and carbon determinations in the crude ash, and state percentages as carbon-free ash. Even with the greatest care, employing the official method for the prepara-

tion of the ash, a lower percentage of carbon-free ash will be obtained than that actually contained in the substance, owing to losses of chlorids and probably more or less phosphorus and sulphur by volatilization. Of the correctness of this fact I am convinced by investigations which have been made by me and published in my inaugural dissertation under the title, "Eine neue Methode der Aschenbestimmung." This fact is also confirmed, as shown in Table I, by the work of Messrs. Moore, Fraps, and Gamble. The average percentage of carbon-free ash, determined in sample No. 1 by what I have called a new method, is from 0.13 to 0.83 per cent higher than the averages determined in the same sample by the official method. Why should the individual percentages of carbon-free ash in the same substance and determined by the same chemist with undoubted care vary all the way from 5.01 to 6.4 per cent? Again, why should the averages of carbon-free ash determined in the one substance by different chemists working equally carefully vary from 5.55 to 6.25 per cent? These variations, I believe, are due to volatilization of varying quantities of certain ash constituents. By the new method, which employs a closed platinum apparatus and calcium acetate solution, individual determinations of percentages of carbon-free ash in the same substance are concordant to the first decimal, as shown by determinations 6, 7, and 8 in Table I.

TABLE II.—*Crude silica in carbon-free ash.*

Analyst.	Determinations.	Percentage in ash from sample No. 1.		Percentage in sample No. 2.	
		Single determination.	Average.	Single determination.	Average.
Moore, C. C.	1	14.13	14.125	15.27	15.23
	2	14.12		15.19	
Fraps, G. S.	1	15.81	15.84	15.44	15.56
	2	15.89		15.33	
	3	15.82		15.91	
Gamble, Wm. P.	1	15.93	19.58	15.50	15.39
	2	26.72		15.34	
	3	16.08		15.35	
	4	15.35			
	5	15.54	15.45		¹ 15.22
	6			15.24	
	7			15.23	
	8			15.19	

¹ These determinations were made in an ash prepared from sample No. 1, but the ash was prepared by Mr. Gamble exactly as in sample No. 2.

Heat employed in the reduction of substances to ash very readily causes more or less fusion, whereby the bases of the ash enter into chemical combination with the silica forming a product or products which resist the action of hydrochloric acid, and are therefore collected and weighed in ash analysis as silica. These circumstances may and do frequently result in very erroneous silica determinations. Take, for example, Mr. Gamble's second determination, 26.72 per cent, Table II, as compared with the other determinations of crude silica. It is 10.64 per cent greater than the next highest. This difference, I am confident, is not due to errors in work, but to fusion with the formation of products not decomposable in hydrochloric acid. In preparing the ash (Tables I and II, Gamble) from which this silica was separated the heat rose a little above dull redness, which immediately caused distinct fusion in the ash.

Sample No. 2 is an ash prepared by the use of calcium acetate in my closed platinum apparatus. The use of calcium acetate solution in moistening the substance

before reducing it to an ash appears to prevent the formation of any trace of indecomposable products in the ash. Consequently, it was expected that not only would the individual determinations of crude silica in sample No. 2 be more uniform, but that the average percentage would be lower than in the ash prepared from sample No. 1 by the official method. With the exception that one chemist found more silica in sample No. 2 than in the ash of sample No. 1, the figures of Table II justify the above expectations.

TABLE III.—*Potash (K_2O) in carbon-free ash.*

Analyst.	Determinations.	Percentage in ash from sample No. 1.		Percentage in sample No. 2.	
		Single determination.	Average.	Single determination.	Average.
Moore, C. C	1	28.27	28.19	30.87	30.96
	2	28.10		31.05	
Fraps, G. S.	1	32.46	32.51	32.09	
	2	32.85			
	3	32.22			
Gamble, Wm. P	1	31.59	29.57	32.19	32.25
	2	27.72		32.27	
	3	29.41		32.29	
	4	31.21	31.09		
	5	30.96			
	6			32.75	32.86
	7			32.87	
	8			32.96	

If fusion in the preparation of ash forms indecomposable products, lower percentages of potash might be expected to accompany higher percentages of silica. That this is the case in a number of instances the figures in Tables II and III show. In addition to this tendency of fusion to lower the percentages of potash, volatilization of chlorids in the preparation of an ash acts in the same direction. These two circumstances account for the higher percentages of potash in sample No. 2 than in the ash of sample No. 1. The tendency of fusion and volatilization to lower the percentage of potash is made very evident by Gamble's determination (2). That a somewhat higher temperature than that of dull redness was allowed in preparing that ash, and that more or less fusion occurred in the ash, are facts known to me. Table I shows that Gamble's determination (2) is the lowest percentage of carbon-free ash; Table II shows that it is the highest percentage of crude silica, and Table III that it is the lowest percentage of potash.

All chemists will agree with me that the place to begin improving the method of ash analysis is in the preparation of the ash. I am of the opinion that volatilization and fusion in its preparation are two important sources of error; that the use of calcium acetate solution overcomes the difficulty of fusion, and that the use of a closed platinum apparatus prevents volatilization.¹

In conclusion, allow me, on behalf of this association, to thank those chemists who have furnished me with analytic results, and also allow me to specially mention the heartiness of the cooperation of the associate referee, Dr. G. S. Fraps, of Raleigh, N. C.

The referee recommends the use of calcium acetate and burning ash in some way to prevent volatilization, which does occur, even with great care, by the official method.

¹ See Twenty-fifth Annual Report of the Ontario Agricultural College and Experimental Farm, 1899, Toronto, Ontario, pp. 42-43.

The president called for any papers bearing on the subject of ash determinations, Mr. Fraps presented one, as follows:

REPORT ON LOSS OF SULPHUR IN PREPARING ASH OF PLANTS.

By G. S. FRAPS.

It is becoming generally known that the sulphur contained in an ash does not necessarily represent the sulphur content of the plant. Berthelot¹ states that the determination of phosphorus and sulphur when the plant is burned to an ash is often incorrect, and discusses the conditions theoretically necessary that no loss take place. Dr. Wiley² says, "Unless special precautions be taken, however, a portion of the organic sulphur and phosphorus may escape as volatile compounds during the combustion," and refers to the method of Berthelot and André for determining total sulphur in soils, viz, oxidation in oxygen and passing the vapors over hot alkaline carbonates. S. Bogdonow³ states that the estimation of the sulphur content of a plant by determining the sulphur in the ash is incorrect. He determines the sulphur in the plant preferably by the method of fusing it with caustic potash and potassium nitrate. Comparing his analyses of cereals made by this method with Wolf's tables of ash analyses, he concludes: (1) The sulphur in the ash does not give even an approximate idea as to the sulphur in the plant; (2) Plants contain considerably more sulphur than has been supposed; (3) The sulphuric acid of the soil is of practical importance. It may be added that he found fertilization with sulphates advantageous to certain Russian soils.

The following experiments were made to determine whether or not sulphur is lost during the incineration of vegetable substances. No case was found in which all the sulphur of the plant was contained in the ash. The determination of the sulphur was made by two methods: (1) Ten grams were burned to an ash at as low a temperature as possible, and the sulphur determined in it; (2) Ten grams of substance were burned with the addition of 20 cc of a solution of calcium acetate containing 29.2 grams per liter, as recommended by Mr. A. E. Shuttleworth. The results were as follows:

Sulphur found in plants.

Substance.	Burned with cal- cium acetate.	Alone.	Loss.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Oat straw	0.158	0.151	4
Crimson-clover hay	.173	.137	28
Green rape	.503	.471	6
Wheat bran	.055	.000	100
Corn silage	.098	.082	16
Timothy hay	.085	.076	11
Cotton-seed meal	.222	.071	68
Soy beans.....	.161	.091	58
Linseed meal.....	.091	.038	58

The sulphur lost when the material was burned by itself is from 6 to 100 per cent more than when burned with calcium acetate, and it has not been proved that the calcium acetate retains all the sulphur.

¹ C. r.: 128, 17.

² Principles and Practice of Agricultural Analysis, Vol. III, p. 37.

³ Jour. russ. phy-chem. Ges.: 31, 471.

The differences in the above table may in some cases seem insignificant, but owing to the quantity of material employed it requires 0.0007 gram of barium sulphate to produce a difference of 0.001 per cent. It is plain that the determination of sulphur in an ash prepared in the usual way is far from giving any idea as to the sulphur in the plant. Any conclusions drawn from such analyses are liable to prove erroneous.

The president here called for the report of the committee on nominations, and the following names were presented by the chairman, Mr. Huston, as officers of the association for the ensuing year:

REPORT OF COMMITTEE ON NOMINATIONS.

For president, L. L. Van Slyke, Geneva, N. Y.

For vice-president, H. J. Wheeler, Kingston, R. I.

For secretary, H. W. Wiley, Washington, D. C.

For additional members of the executive committee, W. R. Perkins, of Agricultural College, Mississippi, and F. W. Traphagen, of Bozeman, Mont.

The secretary of the association was instructed to cast the ballot of the association for the several nominees.

The report of the committee on recommendations was then called for, and was presented, as follows:

REPORT OF COMMITTEE ON RECOMMENDATIONS.

SOIL.

(1) It is recommended that determinations of phosphoric acid be made by N/5 nitric acid as well as by N/5 hydrochloric acid.

Adopted.

(2) It is recommended that a 3-mm sieve be adopted where the soil is to be used for determinations requiring 100 grams or more.

Mr. HARTWELL. I would like to say just a word about the reason for recommending a 3-mm sieve. I found that this size was commonly used in Germany, and it was stated to me that sometimes material would not pass a 2-mm sieve which should be subjected to the action of the solvent. Personally I am not particular whether a 2-mm sieve or a 3-mm sieve is used, but I think we should have a larger sieve for this work.

Recommendation adopted.

(3) The referee's report should contain such data as are necessary for converting the percentage of an ingredient into the number of pounds of the same in a given area of soil at the depth sampled.

Adopted.

The motion of Mr. Ewell, that the referee on soils for next year be instructed to consider the adoption of methods for the mechanical analysis of soils and for the statement of the results thereof, was adopted. The second part of the motion, viz, that the referee on soils for the next year be instructed to consider the adoption of a method for the sampling of soils in accordance with the suggestion of Mr. Dyer, was treated separately and was also adopted by vote of the association.

Mr. HUSTON. There was another matter referred to this committee, but for which we have no papers. It was the motion of Mr. Wheeler in regard to the Gooch-Austin method of determining phosphoric acid. The motion is that the method be referred to the referee on phosphoric acid for the ensuing year.

Adopted.

REPORT OF COMMITTEE ON SUBDIVISION OF FOOD AND LIQUOR ADULTERATION.

Resolved, That it be made the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate and report on one or more of the following subjects:

- (1) Meat and fish.
- (2) Fats and oils.
- (3) Cereal products.
- (4) Infants' and invalids' foods.
- (5) Saccharine products, including confectionery.
- (6) Vegetables, canned, dried, or otherwise preserved.
- (7) Tea, coffee, and cocoa.
- (8) Spices and condiments.
- (9) Vinegar.
- (10) Flavoring extracts.
- (11) Fruit products.
- (12) Fermented and distilled liquors.
- (13) Baking powder and baking chemicals.
- (14) Preservatives.
- (15) Dyes.

Mr. PATRICK. I suggest that if the dairy products, butter and oleomargarine, are not to be included under the head of oils, it would be well to specify that. Otherwise there might be a misunderstanding. The referee, not being here, might understand that the oils would include the butter fats, oleomargarine, etc.

Mr. WINTON. The report is certainly a little indefinite in that regard, but perhaps it is sometimes a virtue not to have things too exact. If the analyst were working on lard and olive oil his work would lead him to try the same method on oleomargarine and dairy products; it is very hard to draw a sharp line. I do not think there is any danger of our referees quarreling as to who shall have the privilege of doing the most. However, I appreciate the inconsistency noted and shall be perfectly agreeable to any amendment.

Mr. Patrick's amendment seconded.

Mr. HUSTON. It would be safer if you made the amendment positive instead of negative. Perhaps it would be better to define what the referees must do.

Mr. WINTON. I think that point could be met by adding a word or two in connection with dairy products in another place. That is to say, the referee on dairy products may be instructed to consider the matter of adulteration in connection with his work.

MR. VAN SLYKE. There are in the various States men who are charged with the enforcement of certain laws, and these gentlemen, as some of us have had occasion to know, are very much stirred up when the word oleomargarine or anything of that sort is mentioned in connection with dairy products, and I think we ought to be somewhat cautious about placing oleomargarine on our records as a dairy product. Between us it is all right, but we do not want to put ourselves on record and have these gentlemen point at us and say that the association classes oleomargarine and similar adulterants as dairy products.

The recommendation on subdivision of foods was referred back to the committee for exact statement.

The president then called for the report on foods and feeding stuffs, which was presented by Mr. Krug.

REPORT ON CATTLE FEEDS.

By WILLIAM H. KRUG, *Referee*.

The present report covers the work of two years, and it has been thought best by the referee to consolidate the results obtained on the samples sent out in 1899 and 1900, respectively, as the instructions with reference to the investigation of certain doubtful features in the analytical methods underwent no change. Early in 1899 Mr. Smith, at that time referee, sent out three samples, consisting of wheat, bran, and peas, respectively. Owing to the fact that the sample of peas was reported to contain very little galactan, it was thought advisable, when it was found that no report would be made at the San Francisco meeting, to send out a sample of clover seed and an additional sample of bran. In the tables which accompany this report the bran sent out in 1899 is marked No. I, and that sent out in 1900 No. II. The instructions which accompanied the samples followed the lines of investigation suggested by the referee in 1898. Subsequently both Mr. Smith, the referee, and Mr. C. A. Browne, jr., the associate referee, found that they would not be able to complete the work, and at the request of the president of the association I have undertaken the compilation of the results. In answer to the circular letter sent out by Mr. Smith thirty chemists requested samples, of whom only eight have returned reports.

THE ESTIMATION OF MOISTURE.

It being evident that variations in the amount of moisture found in the samples would seriously affect the results when calculated to a dry basis, the referee for 1899 requested that this determination be made in all samples. The following results have been obtained:

TABLE I.—*Moisture determinations.*

Analyst.	Wheat.	Bran I.	Bran II.	Peas.	Clover seed.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
E. B. Holland, Hatch Experiment Station	11.98	10.20	10.02	10.93	7.05
G. S. Fraps, North Carolina College of Agriculture.. ..		11.41	13.00		
H. N. Grettenburg, Iowa Experiment Station.....	7.60	7.48	10.13	8.65	5.03
C. A. Browne, jr., Pennsylvania State College.....	11.37	9.75	11.52		7.16
C. P. Beistle, Pennsylvania State College	11.44	9.98	12.26		7.77
L. H. Van Wormer, Michigan Experiment Station..	12.32				8.38
John Stewart, Utah Experiment Station	7.14	6.72			
O. W. Knight, Maine Experiment Station	12.59		12.41	11.68	
W. H. Krug, United States Department of Agriculture	12.68	11.47	11.85		10.37

COMMENTS.

G. S. Fraps.—Moisture was determined by the official method in a Caldwell water oven and drying tubes. The temperature in the oven was 98° C.

C. A. Browne, jr.—Determinations made at close of work, two weeks later than those made by Mr. Beistle. Bottles kept corked except when weighing samples for analysis. Samples dried in a bath at the temperature of boiling water.

C. P. Beistle.—Determinations made at beginning of work, immediately after opening bottles. Bran sample very moist.

(Note by referee: The remarks of Mr. Browne and Mr. Beistle apply to Bran No. II and the sample of clover seed.)

John Stewart.—Caldwell's hydrogen drying oven was used and duplicate determinations were made according to the official method.

DISCUSSION OF RESULTS.

Mr. Stewart also determined the moisture in the sample of wheat and in bran No. I by drying twelve hours at 98° C., which temperature is from 1.5° to 2° higher than the boiling point of water, at the station where he is located. He found that by this method the wheat contained 12.68 per cent and the bran No. I, 10.95 per cent of moisture.

The results stated by me were obtained by drying five hours in vacuo at 100° C.

The table shows that with a few exceptions there is absolutely no uniformity in the amount of moisture reported by the different chemists. I believe that this is chiefly due to the fact that the time specified by the method is not sufficient to remove all the moisture. It has been my experience repeatedly that when substances are dried in vacuo at 100° C. they continue to lose weight for about twelve hours, after which time either a slight increase occurs or the weight remains practically constant. I have found this to be true not only of such materials as fodders and cereals, but also of the comparatively thin films obtained in the analysis of tanning materials. It would seem rational to suppose that in the latter case, when a large surface is exposed, the moisture would be driven off very readily, but this does not appear to be so, a small fraction of the water, amounting usually to from 1 to 2 per cent, being retained very stubbornly and requiring from five to seven hours additional drying. It is evident, on the basis of the results shown in the table, that additional work on the determination of moisture is necessary, the purpose of such work being to fix the time limit and the exact temperature. The latter point would appear to be one of little importance, but it must be remembered that the boiling point of water is rather an uncertain quantity, and may vary from 1 to 2 degrees, depending upon the altitude and atmospheric conditions.

THE ESTIMATION OF STARCH.

Acting upon the suggestions made in 1898, the provisional diastase method and the influence which the solubility of the pentosans may have on the final result have been further investigated.

The following results have been obtained in the estimation of starch:

TABLE II.—*Results obtained with the diastase method.*

Analyst.	Wheat.		Bran I.		Bran II.	
	In original material.	Calculated to dry basis.	In original material.	Calculated to dry basis.	In original material.	Calculated to dry basis.
E. B. Holland, Hatch Experiment Station	52.36	59.49	24.61	27.41	28.60	31.78
H. N. Grettenburg, Iowa Experiment Station	125.96	-----	24.83	26.82	120.11	-----
C. A. Browne, jr., Pennsylvania State College.....	58.99	66.55	28.75	31.85	28.99	32.90
C. P. Beistle, Pennsylvania State College.	59.20	66.84	29.83	33.14	27.68	31.41
John Stewart, Utah Experiment Station.	60.50	65.15	-----	-----	-----	-----
O. W. Knight, Maine Experiment Station	56.86	65.04	-----	-----	30.04	34.31
W. H. Krug, U. S. Department of Agriculture	55.71	63.79	27.00	30.49	28.11	31.89
Averages.....	57.27	64.47	27.00	29.94	28.68	32.46

¹ Excluded from the averages.

COMMENTS.

E. B. Holland.—Wheat and bran No. I: Residues practically free from starch. Both solutions gave precipitate with phloroglucin after regular treatment. Malt solution prepared in the old way by allowing to stand overnight. Bran No. II: The copper oxid was collected in sugar tubes and reduced in a current of hydrogen. The residues, examined with iodine under a microscope, were free from starch. The malt solution was prepared as follows: Twenty-five grams of fresh-ground malt were treated overnight with 500 cc of water in a flat liquor flask, which exposed a large surface, filtered, and 40 cc added to the gelatinized starch. Blanks were made on 32 cc, which allowed a direct subtraction of the equivalent dextrose. One of the most prominent chemists in this section, using large quantities of malt, states explicitly that malt should be ground immediately before use, as it rapidly loses strength afterwards.

C. A. Browne, jr.—Bran No. II: Copper oxid filtered in a tube, oxidized in a current of oxygen and weighed as CuO. Directions prescribed in Bulletin 46 were followed except as regards neutralization. After inversion with hydrochloric acid, sodium hydroxid with a drop of phenolphthalein was used instead of sodium carbonate. The troublesome frothing incident to the use of sodium carbonate was thus avoided, and comparative determinations have shown no difference in the results.

C. P. Beistle.—Copper oxid filtered in tube, reduced in a current of hydrogen and weighed as copper.

John Stewart.—Duplicates on the sample of wheat differed by a little more than 1 per cent. The diastatic fermentation continued twenty-four hours, and even then the microscope revealed insignificant traces of starch in some of the residues. Owing to the absence of convenient means of electrolyzing the copper, I used the potassium cyanide method.

O. W. Knight.—The precipitate was dried at 100° and weighed as Cu₂O, the copper being calculated therefrom. Correction was made for the malt used. A portion treated as for the starch determination gave no pentose bodies either in the bran or wheat.

DISCUSSION OF RESULTS.

The results obtained are rather unsatisfactory with reference to the sample of wheat. The most concordant results were obtained on bran No. II. The fact that such satisfactory figures are reported on bran No. II renders it rather difficult to explain the fluctuation noticeable in the samples of wheat. To some extent this may be due to the different methods used for determining the copper, though this could hardly cause a difference of 8 per cent, such as exists between the lowest and highest results reported. I am rather inclined to believe that the trouble lies in the failure to follow the method strictly, especially since I find that in all cases where the analyst has reported duplicates these agree very well, showing that the analyses have been made in a uniform manner. Naturally slight differences in the manner of using this method would not be so apparent in a material containing comparatively little starch, such as a bran, but would be much more in evidence in the analysis of a cereal. Another cause of error is to be sought in the small amount of the original material which enters into the final calculation, this being only 0.12 gram. Thus a variation of 1 milligram in the amount of copper obtained causes a difference of about 0.4 per cent in the amount of starch found. This forcibly demonstrates that extreme accuracy in the application of the method is essential.

Mr. Grettenburg, of the Iowa Experiment Station, reported comparative results obtained by the diastase, Maercker, and salicylic acid methods, which are given in the following table:

TABLE III.—*Results obtained by the diastase, Maercker, and salicylic acid methods.*

Method.	Wheat.	Bran I.	Bran II.	Peas.	Clover seed.
	Starch.	Starch.	Starch.	Starch.	Starch.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Diastase method.....	25.96	24.83	20.11	58.56	22.37
Maercker method.....	32.93	19.98	28.05	50.90	21.49
Salicylic acid method.....	33.01	25.20	26.86	58.49	21.85

In consideration of the fact that both the Maercker and salicylic acid methods uniformly give higher results owing to the hydrolysis of the pentosans present, the data reported here are, on the whole, so conflicting as to permit of no explanation.

Mr. C. A. Browne, jr., investigated the applicability of taka-diastase as a substitute for the malt solution, and obtained the following results on the sample of wheat and on bran No. I.

TABLE IV.—*Results obtained with taka-diastase.*

Material.	In original material.	Calculated to dry basis.
	Starch.	Starch.
	<i>Per cent.</i>	<i>Per cent.</i>
Wheat.....	53.66	60.54
Bran No. I.....	25.27	28.00

The time of digestion in these analyses was twelve hours, and a blank showed no reducing substance. Mr. Browne concludes that taka-diastase is not a suitable substitute for the malt solution.

In 1898 it was suggested by me that the hydrolytic effect of the malt solution on the pentosans present be fully investigated, since at that time there was quite a difference of opinion on this point between chemists. It is unfortunate that this matter has been practically neglected so far as comparative results are concerned, since it can hardly be called a problem difficult of solution.

Mr. Holland reports that a pentosan determination carried out on 78 cc of the solution obtained with bran No. II yielded 0.0065 grams of phloroglucid, a weight too small to admit of the application of the usual formula of calculation.

Mr. Browne, jr., reports that the malt solution dissolved 2.63 per cent of the pentosans present in bran No. II.

The solubility of the pentosans has been thoroughly investigated by me this year. The first object in view was to determine the extent to which the pentosans would be hydrolyzed by the diastase method. For this purpose 3 grams of the sample of wheat, of bran No. I and of bran No. II were gelatinized, treated with 25 cc of malt solution until the residue was free from starch, cooled, made up to 250 cc, filtered, and 100 cc evaporated to a small volume, in which the pentosans were determined in the usual manner. The following results were obtained:

TABLE V.—*Solubility of the pentosans in the estimation of starch by the diastase method.*

Wheat.		Bran I.		Bran II.	
Wt. phloro-glucid.	Pentosans dissolved.	Wt. phloro-glucid.	Pentosans dissolved.	Wt. phloro-glucid.	Pentosans dissolved.
<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>
.0216		.0208		.0204	
.0212		.0215		.0218	
.0214					
.0212					
1.02135	1.208	1.02115	1.191	1.0211	1.186

¹ Average.

A blank determination with the malt solution showed that this contained no pentosans, and the results indicated that no appreciable amount of the pentosans present in the materials examined had been hydrolyzed. It was then thought advisable to investigate the effect of the time element on the solvent action of the malt solution, the data thereby obtained being, furthermore, confirmatory of those just given. Since starch and various sugars, when distilled with 12 per cent of hydrochloric acid, yield small quantities of volatile products precipitable by phloroglucol, it was thought best to work with a material both rich in pentosans and free from starch. For this purpose a considerable quantity of bran was very finely ground, boiled with water until all the starch was gelatinized, cooled to 55° C., and treated with malt solution until all the starch appeared to be converted. It was then washed thoroughly and again treated with the malt solution at 55° C. for three hours. The residue was again thoroughly washed, boiled with 1 per cent hydrochloric acid for about 15 minutes, neutralized with sodium carbonate, and washed with distilled water until the wash water showed no traces of chlorids. The dried residue was free from starch and was found to contain 33.61 per cent of pentosans. Three grams of this bran were treated exactly as previously described. The time of action varied from one-half to two hours.

TABLE VI.—*Effect of time on the solubility of the pentosans in the estimation of starch by the diastase method.*

One-half hour.		One hour.		One and one-half hours.		Two hours.	
Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.
<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>
.0140	-----	.0176	-----	.0117	-----	.0177	-----
.0132	-----	.0156	-----	.0105	-----	.0186	-----
.0144	-----	.0143	-----	.0110	-----	.0203	-----
.0140	-----	.0151	-----		-----	.0193	-----
¹ .0139	-----	¹ .0157	-----	¹ .0111	-----	¹ .0189	-----

¹ Average.

In every case the amount of phloroglucid was too small to permit the application of the usual formula for calculation to pentosans, and the results are sufficient evidence that during the estimation of starch by the diastase method the error caused by the hydrolysis of the pentosans may be safely disregarded. They also indicate that at least a portion of the volatile products precipitated by phloroglucol are directly derived from the conversion products of the starch itself.

Another question which now arose was as to whether the hydrolysis of this small quantity of pentosans is due to the direct action of the diastase or is caused by the water.

To determine this point 3 grams of the same bran, free from starch, were digested exactly as previously described, the addition of 25 cc of malt solution being omitted. The time of action varied from one-half to two hours. The results are given in the following table:

TABLE VII.—*Hydrolysis of the pentosans by the water in the estimation of starch by the diastase method.*

One-half hour.		One hour.		One and one-half hours.		Two hours.	
Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.	Phloroglucid.	Pentosan.
<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>	<i>Gram.</i>	<i>Per cent.</i>
.0068	-----	.0170	-----	.0124	-----	.0137	-----
.0064	-----	.0149	-----	.0161	-----	.0157	-----
.0100	-----	.0138	-----	.0150	-----	.0152	-----
.0144	-----	.0142	-----	.0142	-----	.0125	-----
.0078	-----	.0126	-----	.0121	-----	.0110	-----
.0147	-----	.0108	-----	.0124	-----	.0122	-----
.0138	-----		-----	.0120	-----	.0111	-----
.0149	-----		-----	.0114	-----		-----
¹ .0111	-----	¹ .0144	-----	¹ .0131	-----	¹ .0129	-----

¹ Average.

In every case the amount of phloroglucid obtained was too small to permit calculation to pentosan. While the results just given appear to be somewhat contradictory, as it is reasonable to suppose that the hydrolysis would be additive in proportion to the increase in time, it must also be remembered that in working with such small quantities and with a method designed to determine a somewhat indefinite organic compound there are many sources of error which will affect the results. A comparison of these data with those given in Table VI shows that the hydrolysis of

the pentosans is mainly due to the action of the water at the temperature employed. The data are compared in the following table, the average weights of phloroglucid being used:

TABLE VIII.—*Comparison of hydrolysis of pentosans in the presence and absence of diastase.*

Time.	With diastase, phloroglucid.	Without diastase, phloroglucid.	Hydrolysis due to diastase, phloroglucid.
	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>
One-half hour.....	0.0139	0.0111	0.0028
One hour.....	.0157	.0144	.0013
One and one-half hours.....	.0111	.0131	
Two hours.....	.0189	.0129	.0060

The only conclusion which can be derived from these data is that the hydrolysis of the pentosans, which is mainly due to the action of the water present, is so small as to be a negligible quantity.

THE ESTIMATION OF THE PENTOSANS.

The circular letter sent out by the referee in 1898 requested that the pentosans be determined in the bran. The following results have been obtained:

TABLE IX.—*Results obtained with the phloroglucol method.*

Analyst.	Bran I.		Bran II.	
	In original material, pentosans.	Calculated to dry basis, pentosans.	In original material, pentosans.	Calculated to dry basis, pentosans.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
E. B. Holland, Hatch Experiment Station.....	17.30	19.27	16.52	18.36
G. S. Fraps, North Carolina College of Agriculture....	18.10	20.43	16.05	18.44
C. P. Beistle, Pennsylvania State College.....	17.84	19.82	15.61	17.71
C. A. Browne, jr., Pennsylvania State College.....			15.61	17.71
W. H. Krug, United States Department of Agriculture.	18.41	20.79	16.31	18.50
Averages.....	17.91	20.08	16.02	18.14

COMMENTS OF ANALYSTS.

E. B. Holland.—Bran No. I: The phloroglucid was dried in three periods of four hours, two hours, and three hours, respectively. At the end of the third period all showed a gain over the second weight. Used 0.85 gram phloroglucol with each test and dissolved it in hydrochloric acid at 88°. The phloroglucol used was Merck's chemically pure, still it reacted strongly for diresorcol by test. Bran No. II: Used 0.9 gram phloroglucol with each test. Dissolved it in hydrochloric acid (sp. gr. 1.06) at 80° C. Employed Merck's chemically pure phloroglucol, though it reacted strongly for diresorcol. As our burners were not sufficiently powerful with the gas pressure then available to use gauze tops and accomplish the distillation of the furfural in the desired time, the flasks were set on wire gauze surrounded by the asbestos paper, with a hole in the center, which allowed the heat to strike the bottom of the flask, but prevented it from striking the sides and charring the material.

G. S. Fraps.—It was found better to distill on a wire gauze than in a bath of Rose's

metal. No trouble was experienced from charring when the 30 cc of acid were added all at once, and in such a manner as to wash down the particles adhering to the sides of the flask. I consider it best to distill off 360 cc without regard to the quantity of pentosans present. All of the substance which is precipitated with phloroglucol does not pass into the first 360 cc. In an experiment on bran No. 2, 0.004 gram of phloroglucol was precipitated from a second 360 cc, equivalent to 0.13 per cent pentosan. Drying requires from seven to nine hours in the water oven. It was found impossible to dry to constant weight in three or four hours.

Following are the various determinations made on brans No. 1 and 2, of which the figures appearing opposite my name in Table IX are the average:

Estimation of pentosans with the phloroglucol method.

Determinations.	Bran No. 1.		Bran No. 2.	
	Pentosans in original material.	Per cent calculated to dry basis.	Pentosans in original material.	Per cent calculated to dry basis.
1	18.20	20.54	16.01	18.40
2	17.98	20.29	16.19	18.61
3	18.14	20.47	16.05	18.49
4			16.07	18.47
5			15.83	18.25

The phloroglucol used for Bran No. 1 was pure only on determination 2, in which Merck's phloroglucol free from diresorcol was used. The Merck's reagent was used in determinations 1 and 2 of bran No. 2. It gave no test with acetic anhydrid and sulphuric acid. Determinations 3, 4, and 5, of bran No. 2, were made with a phloroglucol purified as follows: 11 grams of phloroglucol (impure) were added slowly, with stirring, to 300 cc of hydrochloric acid (1.06 sp. gr.) heated in a beaker and stirred well until almost all dissolved. Some impurities may not dissolve. The liquid was poured into 1,200 cc of the same acid (cold) and the diresorcol allowed to crystallize out. It is filtered immediately before using. The volume containing the required amount of phloroglucol is added to the solution containing the furfural. Over 120 cc would hardly be required in any case. I have used the solution above mentioned in the determination of pentosans in a number of fodders and excrements and found it to give the same results as Merck's chemically pure. The difficulty with impure phloroglucol is that the diresorcol is difficultly soluble in the hydrochloric acid, and is left on the filter. The method of purification just described allows the larger part of the diresorcol to crystallize out, and what remains can do no harm, since it is in solution.

C. A. Browne, jr.—Distillation made from 20-ounce Erlenmeyer flasks. No charring of material upon walls of flask.

DISCUSSION OF RESULTS.

The data given in Table IX are in general very gratifying, as they demonstrate the value of the phloroglucol method and its superiority over the phenylhydrazin method. Mr. Holland reported the results obtained by him in detail and I include them at this point, as they appear to explain to some extent the variations occurring in the results.

TABLE X.—*Influence of time upon the amount of pentosans obtained by the phloroglucol method.*

Number.	Bran I.		Bran II.		Filtrate.
	Average time of distillation.	Pentosans in dry matter.	Average time of distillation.	Pentosans in dry matter.	
	Minutes.	Per cent.	Minutes.	Per cent.	
A.....	11½	a 19.82	9½	c 16.63	Clear.
B.....	11½	a 19.58	8½ ⁹ ₁₀	c 16.64	Do.
C.....	13½	b 18.82	8½	d 16.46	Cloudy.
D.....	15	b 18.85	11½	d 16.34	Do.

a Distilled in the morning and stood over night.

b Distilled in the afternoon and stood over night.

c Precipitated solution, stood 20 hours.

d Precipitated solution, stood 15 hours.

These figures bring out an important point, namely, that the method should be more specific as to the time the precipitated distillate must stand before the phloroglucol is filtered off. They indicate that the time should be at least 20 hours, and it is quite probable that the differences found in Table IX are partly due to variations in the time which intervened between the addition of the phloroglucol and the final filtration.

Mr. Fraps obtained on bran No. 1 with a phloroglucol which contained diresorcol 20.54 per cent, 20.47 per cent, and 20.43 per cent of pentosans calculated to dry matter, while with a chemically pure product he obtained 20.29 per cent. On bran No. 2 he obtained with Merck's chemically pure phloroglucol 18.40 per cent and 18.61 per cent of pentosans calculated to dry matter, while with a phloroglucol purified by himself he obtained 18.49, 18.47, and 18.35 per cent.

It appears to be extremely difficult to obtain a phloroglucol quite free from diresorcol in the market, the absolutely pure product being quite expensive. I therefore think that the method of purification employed by Mr. Fraps is worthy of adoption. Mr. Fraps has lately published a paper on the purification of phloroglucol,¹ in which he shows that the solution of phloroglucol as prepared by him gives as good results as Merck's product free from diresorcol. On three solutions of furfural the weights of phloroglucol were:

Comparison of phloroglucol purified according to Fraps with Merck's phloroglucol.

	I.	II.	III.
Purified phloroglucol.....	0.4575	0.5115	0.4647
Merck's, free from diresorcol	0.4620	0.5124	0.4671

The distillate obtained by the phloroglucol method very often contains small quantities of solid fatty acids, which necessarily are a source of error, as they increase the weight of the precipitated phloroglucol. In Bulletin No. 172 of the North Carolina College of Agriculture and Mechanic Arts Mr. Fraps describes the method as used by him and states that he removes this source of error by allowing the distillate to pass through a small filter paper. This is without doubt a valuable improvement.

¹ American Chemical Journal, Vol. XXIV, No. 3, p. 270.

THE ESTIMATION OF GALACTAN.

Very little work has been done during the last two years on the method provisionally adopted in 1898, and the results are so meager that it is not possible to safely draw any deductions from them. They are given in the following table:

TABLE XI.—*Results obtained with the Galactan method.*

Analyst.	Peas.		Clover seed.	
	In original material, Galactan.	Calculated to dry basis, Galactan.	In original material, Galactan.	Calculated to dry basis, Galactan.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
E. B. Holland, Hatch Experiment Station	0.78	0.88	4.47	4.81
C. A. Browne, jr., Pennsylvania State College			4.43	4.78
O. W. Knight, Maine Experiment Station.....	0.48	0.54		
W. H. Krug, United States Department of Agriculture.....			4.29	4.79
Averages.....	0.63	0.71	4.40	4.79

COMMENTS.

E. B. Holland.—Peas: Mucic acid collected in a weighed filter and dried three hours. Clover seed: After heating with ammonium carbonate the macerated filter paper should be washed several times with hot water by decantation. Upon treating with nitric acid the last time, allow it to stand one-half hour or longer, as the bleaching action of the acid, if continued, will often yield a whiter, cleaner product. I should advise, by all means, the use of the tared filter on which to collect the mucic acid, as the limited quantity of wash liquids has much greater action on the precipitate when collected in the small point of the filter than when spread out on the broader surface of the Gooch crucible. The filters should be weighed in every case in light glass-stoppered weighing vessels.

RECOMMENDATIONS.

Phloroglucol method.—It is recommended that—

(a) The fifth line be changed so as to read “regulated so as to distil 30 cc in about ten minutes, the distillate passing through a small filter paper.”

(b) In the sixth line, after “dilute acid,” the following words be added: “added in such a manner as to wash down the particles adhering to the sides of the flask.”

(c) The distillation be continued until the distillate amounts to 360 cc.

(d) In the eighth line the words “free from diresorcol” be changed to “purified if necessary.”

(e) In the fourteenth line the words “over night” be changed to “at least twenty hours.”

(f) The method of purifying the phloroglucol devised by Mr. Fraps be adopted and added to the paragraph entitled “qualitative test of the purity of the phloroglucol.” The method is as follows:

About 300 cc of hydrochloric acid, 1.06 sp. gr., are heated in a beaker and 11 grams commercial phloroglucol added in small quantities at a time, stirring constantly until it has almost entirely dissolved. Some impurities may resist solution, but it is unnecessary to dissolve them. The hot solution is poured into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1,500 cc. It is allowed to stand at least over night—better several days—to allow the diresorcol to crystallize out, and filtered immediately before using. The solution may turn yellow, but this does not interfere with its usefulness. In using it, the volume containing the required amount is added to the distillate from the pentosan.

Galactan method.—It is recommended that in the fourteenth line, after “ammonia,” the following be added: “The filter paper and contents are then washed several times with hot water by decantation, the washings being passed through a filter paper, to which the material is finally transferred and thoroughly washed.”

It is finally recommended that the terms “phloroglucol,” “dirosorcol,” and “furfural” be adopted.

SUGGESTIONS.

It is suggested that—

(a) The present method of determining the moisture in fodders be further investigated, with a view to fixing the time required and the exact temperature at which the determination must be made.

(b) The effect of various methods of distillation on the results obtained by the phloroglucol method be investigated.

(c) The effect of the length of time which the precipitated distillate stands on the amount of phloroglucol obtained be determined.

NOTES ON THE ESTIMATION OF SUGARS BY WEIGHING THE PRECIPITATED CUPROUS OXID AS SUCH AND AS METALLIC COPPER.¹

By O. W. KNIGHT.

In the estimation of sugars it has been the almost universal practice of chemists to reduce the precipitated cuprous oxid to metallic copper before weighing it, with a view of ascertaining whether equally accurate results could not be obtained by weighing the cuprous oxid and calculating it to metallic copper, the following experiment was undertaken:

A fixed amount of Fehling solution was acted on by different amounts of invert sugar in solutions of equal bulk, and the precipitated oxid filtered into a Gooch crucible washed with water and alcohol, dried at 100° to a constant weight, which took about one hour, and weighed. The weight of copper was calculated therefrom. The cuprous oxid was then dissolved in dilute nitric acid filtered into a platinum evaporating dish, the nitric acid evaporated off and replaced by sulphuric acid, and the metallic copper determined by electrolysis. A table showing the comparison of results by these two methods is appended.

Results obtained by weighing the cuprous oxid as such, and as metallic copper.

Solution.	Method No. 1.		Method No. 2.	Difference.
	Weight of cuprous oxid.	Weight of copper as calculated.	Weight of copper as determined	
	Grams.	Grams.	Grams.	Grams.
1.....	.2640	.2344	.2340	.0004
2.....	.2586	.2296	.2294	.0002
3.....	.2504	.2224	.2224	.0000
4.....	.3290	.2922	.2922	.0000
5.....	.3298	.2929	.2929	.0000
6.....	.3850	.3419	.3380	.0039
7.....	.3786	.3362	.3370	— .0008

At this point in the proceedings Mr. Wheeler exhibited to the members of the association a special form of extraction apparatus.

¹ Presented too late to be read at the meeting.

Mr. Fraps then presented the following paper:

NOTES ON THE DETERMINATION OF PENTOSANS AND CRUDE FIBER.

By G. S. FRAPS.

During the past year the writer has done considerable work upon pentosans, and has adopted some modifications of the provisional method of the association that seemed desirable. Some of these modifications have been communicated to the referee, and have been included in his report, but it has seemed best to make them, together with some other points connected with the determination of pentosans and crude fiber, the subject of a further communication. With regard to the pentosans there are several points to be taken up, the first being the method of distillation.

THE METHOD OF DISTILLATION.

In determining pentosans the material is distilled with 100 cc of hydrochloric acid of 1.06 sp. gr., and, as soon as 30 cc has distilled, 30 cc of fresh acid are added. The association does not prescribe the kind of flask to be used for the distillation, or the manner in which the fresh addition of acid is to be made. I have seen inexperienced analysts make the addition of acid by taking the stopper out of the flask and pouring it in. While the statement may not be necessary for advanced workers, it would be safer to require the acid to be added through a separatory funnel.

Whether the acid is added slowly or all at once is also of some importance. The ebullition of the liquid throws part of the material up on the sides of the flask, especially when it is rich in crude fiber, where it is to some extent withdrawn from the action of the acid, and is also in danger of being charred. If the acid is added in such a manner as to wash these particles down into the liquid, the quantity washed up again during each interval will be less and less, until this precaution is no longer necessary. The acid must be added all at once, or this object can not be accomplished.

THE QUALITY OF THE PHLOROGLUCOL.

It is safe to say that if a phloroglucol free from diresorcol (costing about \$16 per ounce) must be used for the determination of pentosans, very few determinations will be made by the chemists of this country. The writer has very good reason to believe that the method of purification devised at the North Carolina Station, and recommended by the referee for adoption, will be found satisfactory. The theory of this method of purification is very simple. The principal impurity of commercial phloroglucol is diresorcol, which is insoluble in hydrochloric acid of 1.06 sp. gr., and is accordingly precipitated with the phloroglucid precipitate. The method of purification is to dissolve the phloroglucol in hot acid of this strength; part of the diresorcol does not dissolve, part of that in solution crystallizes out on cooling. The solution of phloroglucol in hydrochloric acid will keep for some weeks, but not indefinitely. If kept too long the results will be too high. A solution preserved in the laboratory for about eight months gave results about 0.2 per cent too high.

PRODUCTS OF THE DISTILLATION OF PENTOSANS WITH HYDROCHLORIC ACID.

It is assumed that when materials containing pentosans are distilled with hydrochloric acid of the proper density, the only product which is afterwards precipitated with phloroglucol is furfural. This, however, is not the case, as the following experiments show:

Freshly distilled furfural was dissolved in hydrochloric acid of 1.06 sp. gr., three portions taken, and one distilled in the usual way, until the volume of the distillate was 270 cc. The distillate was made up to 500 cc, and precipitated with phloroglucol as usual. The undistilled portions were precipitated in the same way. The results were as follows:

Precipitates from distilled and undistilled portions of fresh furfural.

Phloroglucid.	Weight of precipitates.		Mean.
	Gram.	Gram.	Gram.
Undistilled portion.....	0.2815	0.2805	0.2810
Distilled portion	0.2804		0.2804
Loss			0.0006

Furfural, therefore, suffers no appreciable loss by distillation with hydrochloric acid. The solution of furfural in hydrochloric acid became brown after standing four days, probably by oxidation. Five portions of the brown solution were taken, and three distilled as just described. The results of these experiments were as follows:

Precipitates from distilled and undistilled portions of furfural after standing.

Phloroglucid.	Weight of precipitates.			Mean.
	Gram.	Gram.	Gram.	Gram.
Undistilled portions	0.2880	0.2863		0.2871
Distilled portions	0.2785	0.2779	0.2762	0.2775
Loss.....				0.0096

When the furfural is exposed to the air for four days and distilled with hydrochloric acid it loses 3.2 per cent in weight.

Now, when vegetable materials were distilled with hydrochloric acid in the usual way for the determination of pentosans, and the distillates divided into two portions and one portion distilled as described above, and the other precipitated directly, the differences were always found to be greater than 3.2 per cent. Some body, which, unlike furfural, is easily affected by hydrochloric acid, must be present in these distillates. The quantity of this body is different for different materials, as may be seen from the following tables:

Distillates stood over night before precipitation or redistillation.

Materials.	Loss.
	<i>Per cent.</i>
Cotton-seed meal.....	7.4
Corn silage and corn bran.....	15.6
Cowpea meal.....	17.9
Crimson clover hay	11.7
Irish potato vines	10.2
Corn bran	22.6

Distillates precipitated or distilled the same day.

Materials.	Loss.
	<i>Per cent.</i>
Pine straw.....	13.2
Oxycellulose	13.8
Gum arabic.....	12.9
Corn cobs.....	10.1
Okra hulls.....	11.5
Oak sawdust	6.9

These figures go to show that when vegetable materials are distilled with hydrochloric acid there is formed, besides furfural, one or more bodies which are easily changed by the hydrochloric acid. This investigation is still in progress, and will be pushed to completion when possible.

Before passing on, it should be noted that when the distillate stands for a time usually a black precipitate settles out in small quantity. This was always filtered out before working on the solution.

NOMENCLATURE.

Some of the terms used by the association in the description of the pentosan determination are not in accord with the best terminology. The ending "ol" has been adopted generally to designate the alcoholic or phenolic character of a body. For this reason the term furfural is objectionable, since the body in question is not an alcohol or a phenol but an aldehyde. Either "furfural" or "furfuraldehyde" would be better. Both are in use. The substances which are called phloroglucin and diresorcin are phenolic in character and should terminate in "ol." Making the same change which converts glycerin to glycerol, we change these names to phloroglucol and diresorcol.

The term pentosan is not fully justified in the light of our present knowledge. We assume that all the furfural obtained by distilling vegetable materials with hydrochloric acid is derived from pentosans, meaning thereby a body $(C_5H_8O_4)_x$ which would be analogous to the hexosans starch $(C_6H_{10}O_5)_x$ or cellulose $(C_6H_{10}O_5)_x$. But it is well known that oxyhexoses will yield pentosans under the same conditions, examples being glycuronic acid $(COH.(CHOH)_4COOH)$, osones, $(CHOH(CHOH)_3CO.COH)$, and oxycelluloses, which is cellulose in which one or more of the hexose groups has been oxidized to groups like glycuronic acid or osones.

Cross and Bevan propose to call the furfural-yielding substances furfuroids (furfuran-like). A better term, it seems to me, would be pentosoid—meaning pentose-like—and these bodies are like pentoses in that they yield furfural.

CRUDE FIBER.

When one attempts to introduce the determination of pentosans into his scheme of analysis for feeding stuffs he meets with difficulty, in that crude fiber usually contains pentosans. Crude fiber may contain 40 per cent of the pentosans present, and often contains 20 per cent. For example, 14.4 per cent of the crude fiber of timothy hay is pentosans, and 20.5 per cent of the pentosans are contained in the crude fiber; 13.4 per cent of the crude fiber of crab-grass hay is composed of pentosans, and 19.6 per cent of the pentosans are in the crude fiber. Evidently pentosan can not be subtracted from nitrogen-free extract, and the only way to place this determination in a systematic analysis of feeding stuffs is to determine it in the crude fiber also, that is if we retain our present method for crude fiber. The pentosans in the crude fiber are different from the pentosans in the nitrogen-free extract, for they are digested to a less extent than the latter or the crude fiber. In spite of this such a separation is entirely arbitrary, and it would be better to adopt a method for the crude fiber determination in which the pentosans are dissolved completely. Such a method has been devised by J. König [Analyst, **23**, 47 (abstract)], and is as follows:

Three grams air-dried substance are placed in a porcelain basin with 200 cc glycerol of 1.23 sp. gr., containing 20 grams concentrated sulphuric acid per liter. The basin is placed in an autoclave and steamed for 1 hour at 137° C. It is allowed to cool to 80°–100° C, 200–250 cc water added and the contents filtered while hot, washed, and ignited as usual. In the absence of an autoclave, the digestion may be carried out in a 600 cc flask connected with a reflux condenser. The glycerol is brought to a very gentle boil, boiled 1 hour, and finished as before.

The crude fiber prepared by this method is practically free from pentosans. Kellner, Herring, and Zähn¹ conclude from their study of this method that it is an improvement over the laborious method now in use, and recommend it for use in digestion and metabolism experiments as being more accurate, more rapid, and more easily carried out than the present method, and as effecting a sharper separation of constituents.

Mr. Wheeler presented the following resolution and requested that the same be transmitted as soon as possible, so that it might be read before adjournment:

Resolved, That the thanks of the association be extended to the National Grange Patrons of Husbandry for the several courtesies extended to this association.

Motion carried.

The president then called for the report on dairy products, and the referee being absent the report was read by Mr. Frear, as follows:

REPORT ON DAIRY PRODUCTS.

By J. B. WEEMS, *Referee*, and F. W. WOLL, *Associate Referee*.

The time for the investigation in connection with the work of the association being so short during 1899, it was decided to continue the work of last year and in addition to investigate other methods for the determination of casein and albumin, of interest in this connection.

In preparation for the work circular letters were sent to all chemists connected with experiment stations, asking their cooperation in the work of the year. The result of these requests was that there were received twelve favorable replies, and to each of the chemists offering to aid in the work a sample of skim milk preserved with formaldehyde was sent with the following circular:

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

METHOD OF ANALYSIS OF DAIRY PRODUCTS.

Your cooperation is respectfully requested in a study of the following methods for the determination of casein and albumin in milk:

- I. (a) *Casein, official method*:
Determine the amount of casein in the sample of milk as given under "Official method of determination of casein in cow's milk," p. 55, par. 1, Bul. 46, rev. edition, Division of Chemistry, Dept. of Agriculture.
- (b) *Albumin, provisional method*:
Determine the albumin in the filtrate of the above, as outlined in "Provisional method of the determination of albumin in milk," p. 55, par. 2, Bul. 46, rev. edition.
- II. (a) *Casein, official optional method*:
Determine the casein as given in the "Official optional method for determining casein in milk," p. 55, par. 3, Bul. 46, rev. edition.
- (b) *Albumin, provisional optional method*.
Determine albumin in the filtrate of the above method as given in par. 4, "Provisional optional method for determining albumin in milk," p. 55, Bul. 46, rev. edition.
- III. (a) *Casein, modification of official method*:
Carry the method through as described, to and including the washing of the precipitate, then neutralize filtrate as nearly as possible with sodium hydroxid, filter off any precipitate that may form and add it to the former precipitate for the nitrogen determination.
- (b) *Albumin, modification of provisional method*:
To the filtrate from the preceding determination add 10 cc of 20 per cent tannin solution; heat, filter, wash with water, and determine nitrogen.

¹ Exp. Station Record (abstract), XI, 705.

IV. (a) *Casein, alum method:*

To 10 cc of milk add 50 cc of water at 40°. Then add 1.5 cc of alum solution saturated at 40° or higher. If the precipitate fails to settle promptly add 0.5 cc more of the alum solution; filter, wash with water, and determine the nitrogen.

(b) *Albumin, modification of provisional method:*

To the filtrate from the preceding determination add 10 cc of 20 per cent tannin solution; heat, filter, wash with water, and determine nitrogen in the usual manner.

V. *Indirect, or Penny's method:*

(a) Dilute 25 cc of milk with 150 cc of distilled water in a 250 cc measuring flask. Then add 5 cc of alum solution (see above, IV (a)) to precipitate the casein, and fill to the mark. Determine the nitrogen in 100 cc of the filtrate in the usual manner.

(b) Dilute 25 cc of milk with 150 cc of distilled water in a 250 cc measuring flask. Then add 25 cc of 20 per cent tannin solution to precipitate the casein and albumin; heat, cool, fill the flask to the mark with the distilled water and filter. Determine the amount of nitrogen in 100 cc of the filtrate.

(c) Determine total nitrogen in the sample of milk by the Kjeldahl method. Multiply the amount of nitrogen found in *a*, *b*, and *c* by 6.25, and from these results calculate the percentages of casein and albumin in the sample of milk.

In reporting results give separate figures for *a*, *b*, and *c*.

In reporting the analytical results for the above methods please give all the data and information that will be of interest. We shall be glad to have you state which methods you regard most favorably, and the considerations or reasons for your opinion, as well as any objections which you may find to any of the methods.

Yours, very respectfully,

J. B. WEEMS, *Referee*.

F. W. WOLL, *Associate Referee*.

The results obtained by the chemists cooperating in the work were as follows:

Results of the comparison of methods for the determination of casein and albumin in milk, 1900.

Analyst.	Casein.					Albumin.					Total nitrogen × 6.25.
	I.	II.	III.	IV.	V.	I.	II.	III.	IV.	V.	
	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
Department of feeds and feeding, Massachusetts Experiment Station.....	2.76	3.02	2.78	2.69	2.67	0.18	0.31	0.51	0.59	10.02	3.42
C. B. Cady, Missouri	2.69	2.88	2.84	2.65	2.65	.32	.28		.60	.61	3.31
H. A. Huston and A. H. Bryan, Indiana	2.78	2.83	2.85	2.83	2.81	.27	.45	.48	.50	.46	3.39
John White and R. W. Thatcher, Nebraska.....	2.80	2.67	2.80	2.65	2.54	.14	.10	.31	.49	.59	3.26
C. L. Penny, Delaware.....	2.68		2.77	2.68	2.68	.18		.35	.49		3.30
J. A. LeClerc, Geneva, N. Y.	2.71	2.84	2.72	2.71	2.69	.30	.32	.44	.53	.49	3.35
T. M. Price, Maryland	2.42	2.71	2.63	12.32	2.45	.38	.43	.53	.44	.53	3.15
F. W. Woll and Alfred Vivian, Wisconsin	2.67	2.68	2.66	2.71	2.48	.18	.29	.36	.35	.59	3.27
C. E. Ellis, Iowa.....	2.65	2.74	2.72	2.64	2.64	.22	.27	.47	.44	.58	3.22
C. E. Gray, Iowa	2.74	2.70	2.75	2.71	2.54	.31	.30	.48	.61	.54	3.27
Average	2.690	2.756	2.752	2.697	2.615	.248	.306	.437	.504	.549	3.294
Highest	2.80	3.02	2.85	2.83	2.81	.38	.45	.53	.61	.61	3.42
Lowest	2.42	2.67	2.63	2.32	2.45	.14	.10	.31	.35	.02	3.15

¹Omitted in average.

The department of feeds and feeding of the Massachusetts Experiment Station makes the following remarks upon the work:

The official optional method for casein proved objectionable because of the slow filtration and difficult washing of the precipitate, due to the crystallizing out of the magnesium sulphate on the filter as the solution cooled.

In the alum method undoubtedly the addition of 2 cc of alum would insure perfect precipitation and a clear filtrate, which was not obtained in every case with 1.5 cc.

In the direct method 5 cc of alum did not yield a clear filtrate and the filtration was exceedingly slow. Again, in the indirect method 25 cc of tannin solution apparently did not precipitate all the casein and albumin, judging from the results, though the filtrate was clear.

After considering both the manipulation of the several methods and the results obtained thereby we should be inclined to indorse the modified official method for casein without a second filtration after neutralizing the soda, and the modified provisional method for albumin.

Mr. Cady's experience with the methods is shown in his conclusions:

In "Section III, Modification of Official Method," after neutralizing the filtrate from the acetic-acid precipitate with sodium hydrate, I obtained a somewhat gelatinous precipitate, which filtered with much difficulty, giving very little albumin when treated as directed.

The tannic acid which I used was marked C. P. However, on making blank determinations, using 10 cc of the 20 per cent solution, I found that 10 cc of the solution contained 0.0054 gm of nitrogen, and acting on the supposition that all of the nitrogen contained in the tannin solution was retained in the filtrate, I made reduction accordingly.

Mr. H. A. Huston drew conclusions as follows regarding the methods:

The magnesium-sulphate method has always proved very tedious in our hands, while the method of Penny has the merit of avoiding long filtrations and washings, although requiring two extra nitrogen determinations.

Mr. John White reports as follows regarding the methods:

The official method for casein appears to be the most satisfactory one. It is rapid, easy of manipulation, and gives very concordant results. The suggested modification of this method, viz, the neutralization of the filtrate by sodium-hydrate solution and refiltration, appears to be of little value, since no appreciable precipitate is formed on neutralization.

The alum method is also a favorable one for casein. It gives somewhat lower results than the precipitation by acetic acid, but otherwise is quite satisfactory.

The official optional method does not appear to be satisfactory. It would seem from observations made that the proportion of nitrogenous bodies precipitated by magnesium sulphate varies between limits too wide to justify the use of this precipitant. Further, the use of a substance which crystallizes so readily as does magnesium sulphate in a solution saturated at a temperature above the working temperature of the laboratory causes obvious difficulties in manipulation which are hard to overcome.

Concerning the methods for the precipitation of albumin, tannic acid seems to be by far the most satisfactory precipitant. It yields somewhat higher results than does acetic acid, but the results are very concordant, the differences in the percentages obtained from the different filtrates being apparently due to differences in the proportion of nitrogenous bodies precipitated as casein by the various precipitants used.

The indirect or "Penny's" method is rather a neat one and easy of manipulation. It does not, however, appear to possess any particular advantage over the direct method and is not quite so rapid, more nitrogen determinations being required.

Mr. LeClerc gives the results of his experience in the work in the following comments:

Method II (a).—Required six hours to filter and wash. Magnesium sulphate will precipitate any globulin that may be in milk. The washing takes so long that possibly the precipitate is not absolutely free from albumin. Hence a slightly higher result for casein.

Method III (a).—I was unable to obtain a precipitate on neutralization. Got only a slight cloudiness, which was not removed even after repeated filterings.

Method IV (a).—Obtained good precipitations, but found it necessary to have the solution, after adding the water, at least 40° C. Thorough washing of the precipitate

to remove all traces of alum is necessary, because the alum sometimes contains ammonium as impurity.

Method V (a).—This method works very well, provided precipitation is done at about 42° C. and care is taken that the alum is not a mixture of potash and ammonium alum. I had much difficulty with this method till I found that my alum contained an ammonium salt.

I prefer Methods I (a) and IV (a).

It has been shown in this laboratory by Van Slyke and Hart that milk generally contains nitrogen compounds other than casein and albumin, and that these bodies are thrown down by tannin. Hence higher results in all cases where tannin was used as a precipitate for albumin.

Mr. C. L. Penny makes the following comments on the results obtained in carrying on the line of work outlined by the referee:

I (a).—I find acetic acid a good precipitant and convenient to use.

(b).—Filtration slow in many cases, but for the most part the method worked well in the execution. The results, 0.183 per cent, were far too low, being about 30 per cent of the true result.

II (a) and (b).—The filtration was exceedingly slow, so slow, in fact, that this method was abandoned; hence I have no analytical results. My impression is that Dr. Van Slyke had a similar experience with the magnesium-sulphate precipitation. I can not find the reference now; it was probably a private communication. At all events, magnesium sulphate did not seem to promise enough to justify the expenditure of more time.

III (a).—The neutralization of the filtrate, contrary to my former experience as reported, does give an additional precipitate, amounting in this case to about one-tenth of 1 per cent, reckoned on the original sample. The second filtration I found in one case fairly rapid, in two other cases very slow, so slow that I abandoned that part of the work. I think a double filtration is hardly practicable.

(b).—The tannin filtration was fairly rapid, but not very clear. The result was far too low, but little over half of the true one.

IV (a).—Alum seems to me to be the best precipitant. The precipitate is the easiest and quickest to wash. The several results are very concordant. I think that alum furnishes as good a practical reagent for separating albumin from casein as any that I know of.

(b).—The tannin here gave a good precipitate and a clear filtrate. The filtration was rapid. The result was nearer the truth for albumin than that given by any other precipitant. The several results are also concordant.

V (a).—There is little more to be said about alum than has been said above, so far as my experience goes. The precipitation was good, the filtration rapid, and the filtrate clear. I evaporated 100 cc in Kjeldahl flasks after adding 30 cc of strong sulphuric acid over a slow flame, in some cases with an air blast and in some cases without. I would prefer 50 cc (representing 5 cc of milk) to 100 cc on account of the time spent in reducing the volume of the solution. An air blast facilitates this part of the operation, however, greatly. With 50 cc of filtrate the solution in the Kjeldahl flask may be brought down, by careful adjustment of the flame, to a point where there is no danger of foaming. Thus I prefer 50 cc to 100 cc, and I believe it will be quite accurate enough, for the nitrogen determinations in milk agree much more closely in my experience than those of most other bodies.

(b).—The filtrate from tannin in my work showed 0.044 per cent of nitrogen or 0.277 per cent of protein. But the tannin solution itself contained, in the quantity used to precipitate nitrogen equivalent to 0.225 per cent of protein, i. e., had the milk sample been free from all nitrogen, the tannin alone would have shown 0.225 per cent of protein. Hence this test falls out as entirely worthless, as I have made it, and I therefore omit its results from the table, as it shows absolutely nothing.

(c).—The total nitrogen was determined in 10 cc of milk directly in the usual way by the Kjeldahl method.

Supplementary.—I precipitated 25 cc of milk in the 250 cc flask with acetic acid (10 per cent), 4 cc as nearly as possible according to the official method for casein, I (a), so far as dilution and temperature were concerned; but necessarily the dilution was somewhat less, viz, 150 cc of water, or sevenfold instead of tenfold, as in the official method, for casein. The filtration was speedy and the filtrate clear; the precipitant seemed to be an excellent one. The result was a trifle lower than by V (a), with alum, viz, 0.58 per cent instead of 0.62 per cent with the latter. As between alum and acetic acid, I have but little preference; if any, it is for alum. Perhaps, however, the alum is the more uniform and the acetic acid the more capricious, the latter depending more on details of treatment than the former.

In conclusion, as to a final choice of methods, I like the method of difference for the following reasons:

- (1) The filtration is exceedingly rapid and there is no washing of the precipitate.
- (2) The total protein is sure to be given accurately by this method, and that, in fairness to the sample, is most important. I fear a shortage in albumin if it be determined by actual precipitation with heat or tannin.
- (3) The difference method uses but a single reagent to precipitate, either alum or acetic acid as may be decided on.
- (4) Of the three quantities, total protein, casein, and albumin (the first of which is practically, if not absolutely, equal to the other two added together), the method of difference determines in any event the one that is by far the easiest, viz, total protein, instead of determining the more difficult two, casein and albumin.

Mr. T. M. Price reports as follows:

I prefer the alum method, as the manipulation is such that it precipitates rapidly and filters very easily, but I find that the alum method seems to give a little lower results than the others. The regular official method is very easy to work and gives no particular trouble. In the optional method the filtration is very slow, and we sometimes had difficulty because of the crystallizing of the magnesium sulphate in the stem of the funnel.

Mr. F. W. Woll makes the following comments:

As to the comparative value of the different methods I may say that my preference is decidedly the alum method or the Penny method, if either is found correct. The precipitated casein in the former method comes down in large clots that allow of rapid filtration. If total nitrogen, casein, and albumin are to be determined in a sample of milk, the Penny method would be very convenient, provided we can depend on the results. In Method III no precipitate of casein was obtained by neutralizing the filtrate with soda solution, but you will notice that the albumin was increased. The per cent of casein in Methods I and II ought, therefore, to have been the same.

In comparing the results obtained from the sample of milk it will be noticed that the average for Method I, or the official method, was 2.69 per cent, and for Method IV, or the alum method, was 2.697 per cent, a difference of 0.007 per cent. After considering these results and the reports by the analysts, as well as the results obtained in previous years, the reporters feel warranted in making the following recommendation:

(1) That paragraph 3 of Bulletin 46, revised edition, p. 55, entitled "Official optional method for determining casein in milk," "To 5 grams of milk, 50 cc of a solution of magnesium sulphate," etc., to be changed to read as follows:

(3) "*Official optional method for determining casein.* To 10 cc of milk add 50 cc of distilled water at 40°. Then add 2 cc of alum solution saturated at 40° or higher. Allow precipitate to settle, transfer to a filter, and wash. The washed precipitate and filter paper are digested as in the regular Kjeldahl method as outlined in the official method, paragraph 1. Calculate casein by multiplying the amount of nitrogen found, by 6.25.

(2) That paragraph 4 of Bulletin 46, p. 55, entitled "Provisional optional method for determining albumin milk," etc., be changed to read as follows:

"*Provisional optional method for determining albumin in milk.*"—To the filtrate from the preceding add 10 cc of 20 per cent tannin solution, heat, filter, wash with distilled water, and determine nitrogen by the Kjeldahl method. Multiply the result by 6.25 for albumin.

Mr. EWELL. I have a brief note in reference to one of the methods which comes under this head.

REPORT ON THE DETERMINATION OF THE SPECIFIC GRAVITY OF FATS AND OILS AT THE TEMPERATURE OF BOILING WATER.

By E. E. EWELL.

The temperature of boiling water has been a reference temperature much favored by chemists for this determination, because it makes possible a direct comparison of the density of practically all fats, oils, waxes, and free fatty acids of commercial

importance, whether they be liquid or solid, at ordinary temperatures. The official methods of this association have included directions for this determination for several years. In accordance with these directions, the weight of a given volume of fat at the temperature of boiling water is compared with the weight of the same volume of water at the same temperature. Two methods are official for the standardization of the flasks:

(1) The determination of the weight of water contained in the flask, at a temperature of boiling water, by actually filling it at that temperature, cooling to room temperature and weighing.

(2) The determination of the weight of water contained in the flask at any convenient temperature and the use of this result in the calculation of the weight of water held by the flask at 100° C., using for this purpose the recorded data for the coefficients for the expansion of water and glass.

Inasmuch as the directions given in the official methods for this calculation contain an error and the formula given is incomplete, in that it leads only to the volume of water contained in the flask at the desired temperature instead of its weight, I was directed by the Chief of the Division of Chemistry to revise the formula and to inquire into the relative accuracy of the two methods of standardizing the flasks to be used for this determination.

The proper formula was readily arrived at and is as follows:

$$W^T = W^t \frac{d^T}{d^t} [1 + \gamma (T - t)], \text{ wherein:}$$

W^t = weight of water at temperature of observation;

d^t = density of water at temperature of observation;

t = temperature of observation;

W^T = weight of water at the desired temperature;

d^T = density of water at the desired temperature;

T = the temperature for which the weight is to be calculated;

γ = coefficient of cubical expansion of glass.

If $T = 100^\circ$; and

$\gamma = 0.000028$; the formula becomes:

$$W^T = W^t \frac{0.95863}{d^t} [1 + 0.000028(100 - t)].$$

The use of a formula of this kind necessarily involves sources of error of appreciable magnitude. This and certain other considerations named below made it desirable to determine experimentally the variations existing between the results of the two methods in use for standardizing flasks for this determination, and to critically examine the sources of error attending each of them.

The factors tending to produce variations between the results of the two methods may be briefly enumerated as follows:

(1) *The coefficient of cubical expansion of water*, which may be regarded as one of the best determined constants of nature. The values for the density of water at various temperatures, as given in *Landolt & Börnstein's Tabellen*, may be regarded as accurate beyond the requirements of the determination under discussion.

(2) *The coefficient of the cubical expansion of glass*, which is a matter of some doubt. It varies with the chemical composition of different kinds of glass and with the physical changes to which a given kind of glass may be subjected. A value for this coefficient commonly found in text-books is 0.000026. The recorded results for different kinds of glass vary from 0.000017 to 0.0000305. Schulze¹ in the course of his

¹ Alfred Schulze. Ueber die Ausdehnung der wichtigsten Titirflüssigkeiten durch die Wärme. Zeit. anal. Chem., 1882, **21**, 167-177.

experiments on the coefficients of expansion of certain volumetric reagents, found that the coefficient of cubical expansion of the glass of the apparatus in use by him at that time varied from occasional values as low as 0.0000288 to 0.0000305, the most of his results lying between the number last named and 0.0000295. In the second formula which I have tentatively suggested above, and which was used in the calculation of the results that are to follow, I have inserted as the value of this constant in the general formula the number 0.000028 which is a mean value. These numbers have so many ciphers between the significant figures and the decimal point that they seem at the first glance to be of little importance. Move the decimal points two places to the right and we have the expansion of a 100 cc flask stated in fractions of a cubic centimeter for each degree of increase in temperature. Move the decimal points two more places to the right and we have the expression for the increase in capacity of a 100 cc flask when warmed from 0° to 100° C.; 0.170 and 0.305 cc. These numbers are about one and one-fourth times as great as the expansion which has to be corrected for in standardizing a 100 cc specific gravity flask for use at 100° by calculation from the results of weighings made of the water contained in the flask at ordinary temperature. The highest and lowest numbers differ by 0.135 cc, which is approximately equal to the same fraction of a gram. Such an error in the standardization of a flask would entail an error of at least one unit in the third decimal place of the result of a specific gravity determination. With proper apparatus and manipulations the error should very rarely or never pass to the left of the fifth decimal place of the result. I trust that this discussion sets forth clearly the necessity of having our specific gravity flasks made of glass of which the coefficient of cubical expansion is known with precision to the sixth decimal place at least, if we are going to standardize them for use at high temperatures by calculations based on experimental results obtained at low temperatures.

(3) *Errors attending the determination of the temperatures of observation:*

(a) At the temperature of the laboratory this may readily be done by means of a suitable thermometer to within 0.1°. Only a verified thermometer should be used for this work. Working at substantially the temperature of the air, the correction to be applied to the reading of the thermometers for the unimmersed portion of the column of mercury is practically zero. One-tenth of a degree of temperature means at 25° a change in the density of water of 0.000025 and approximately the same error in the result of the determination of the specific gravity of another liquid.

(b) The determination of the temperature of observation at or near the temperatures of boiling water involves considerably greater difficulties. If we undertake to ascertain the actual temperature by means of a thermometer, we find it necessary to apply a correction for the unimmersed portion of the mercury column of the thermometer, that may in some cases be as great as one whole degree. I doubt if this correction can be ascertained within less than 0.2 or 0.3 of a degree, involving in the latter case an error in a density determination of about two units in the fourth decimal place (0.00024). It is therefore evident that it is necessary to use a short thermometer or a form of apparatus that will permit the immersion of the thermometer to the top of the column of mercury, or very nearly to that point.

If we undertake to fix the temperature by means of boiling distilled water, we are in still greater trouble. In the first place, it is not as easy as might at first be supposed to heat water to exactly 100°, or even sufficiently near for our present purpose. We have a mass of water inclosed in a vessel whose sides are constantly radiating or conducting away heat while it is being agitated by bubbles of steam, having a temperature of the boiling point of the water or slightly more. Unless the sides of the vessel are well insulated, or are heated to or above 100° throughout their entire surface, the temperature of the water may easily be some tenths of a degree below that of the vapor it is giving off. The temperature of boiling water is, therefore,

dependent upon the nature of the vessel and the vigor of the boiling. In this connection the statement made by Allen¹ is interesting:

"It will be observed that the densities in the third column of the foregoing table are recorded as obtained at a temperature of 98° to 99° C. In the author's laboratory water ordinarily boils at 99° C., and oil immersed in a vessel of boiling water rarely reaches a temperature exceeding 98.5° C."

Now, this learned author, for whom we all have such great reverence, does not mention the influence of atmospheric pressure on the boiling point of water, and fails to state whether correction was made for the column of the thermometer not immersed in the water. In the absence of direct statements we must, of course, credit him with having made proper corrections. I have mentioned above that the correction may approach one whole degree. Allen, however, uses only 3 and 4 decimal places in stating the results given in the table mentioned in the quotation.

The matter of atmospheric pressure is to my mind the insurmountable obstacle to the use of the boiling point of water for a reference temperature for the purpose under consideration. This is certainly true when the weight of fat at the boiling point of water is compared with the weight of the same volume of water at some lower temperature, 4°, 15.5°, etc., having been proposed for this purpose. In the official method water and fat are both compared at the temperature of boiling water, and in this way the influence of variations in this temperature are eliminated in a great measure. Allen (in the table of results mentioned above) gives the specific gravities of certain oils at 15.5° C. and at 98° to 99° C., water at 15.5° C. being 1,000 in each case. Results obtained by the method of the Association of Official Agricultural Chemists can not be readily compared with results stated in the manner adopted by Allen, since the temperature of the boiling point of water may vary between comparatively wide limits. During the series of experiments reported below the barometer reading varied from 753 to 767 mm. This variation of 14 mm represents a variation in the boiling point of water of approximately 0.5° C. When we recall that at Charleston, South Carolina, and other tide-water points the mean boiling point of water is approximately 100° and sometimes more, and at Denver, Colorado, it may fall as low as 95°, the irrationality of the method is apparent. It would seem to be more scientific to direct that the specific gravities of fats and oils be determined at exactly 100°, and that the results be stated in terms of—that is, compared with—water at the same temperature. This would involve, however, the difficulty that it would be necessary to standardize the flasks by calculation in all cases in which the boiling point of water falls below 100°. For example, in high altitudes the water would begin to boil in the flasks several degrees below 100° C. It would rarely be necessary to correct by calculation for more than 5° C.

(4) *Errors attending the manipulation of filling the flask* are relatively smaller the larger the flask. These become greater as the temperature is raised, however, and are undoubtedly considerably greater at 100° than at ordinary temperatures. Evaporation is greater when the weighings of water are being made, and there is difficulty in having the stoppers at the proper temperature at the time of their insertion in the flask.

(5) *Fatigue dilatation*.—This term is used to designate a well-known phenomenon attending the heating and cooling of glass, viz, the tardiness with which a glass vessel returns to its original size when cooled to the ordinary temperature after having been heated to a higher temperature. The extent of the fatigue dilatation varies with different kinds of glass and is well illustrated by the well-known depression of the zero point of a thermometer immediately following the heating of the thermometer bulb to a high temperature.

The amount of fatigue dilatation exhibited by three specific-gravity flasks made of ordinary Bohemian glass is shown in the table of results given below. The flasks

¹ Commercial Organic Analysis, Leffmann's edition, Vol. II, Pt. I, p. 31, footnote.

had not been used for several months previous to the experiments made on November 12 and 13. On November 15, within less than an hour after the flasks had been heated for one-half hour at the boiling point of water, weighings were made to determine the capacity of each of the flasks at room temperature. The results are given in the table, and when the calculated capacities of the flasks at 100° are compared with the mean of those calculated from the results of the four series of determinations made on November 12 and 13, it will be seen that the capacities on November 15 were 0.0022, 0.0033 and 0.0035 gram, respectively, higher than the mean of the results based on weighings made on November 12 and 13, i. e., before the flasks had been heated. These results indicate that, unless we use flasks made of glass exhibiting very small fatigue dilatation, this factor must be considered when we desire results of the highest accuracy.

The error in the result of a specific-gravity determination arriving from a given error in the determination of the capacity of the flask may be readily ascertained by reference to the following table:

Error in specific gravity resulting from given error in weighing flask.

Size of specific-gravity flask.	Error in weight necessary to cause errors in specific gravity, results of—		
	0.001	0.0001	0.00001
	Gram.	Gram.	Gram.
25 cc.....	0.0250	0.0025	0.00025
50 cc.....	0.0500	0.0050	0.0005
100 cc.....	0.1000	0.0100	0.0010

In this connection it is proper to consider certain hypercriticisms that have been made from time to time of methods of analysis in general use. I refer to those methods which direct that a solution be prepared by boiling the substance with a solvent in a graduated flask, cooling and diluting to the mark in the same flask. It has frequently been stated by chemists, who apparently have not attempted to ascertain the amount of the attendant error, that this practice is bad; that the flask does not return to its original capacity on cooling, and consequently an error is introduced. The results cited above indicate that for glass of the quality used in the experiments reported this error for a 1-liter flask would amount, approximately, to from 0.066 to 0.088 cc. This is an error that certainly may be neglected in all ordinary routine analytical work. An estimate of the error can be arrived at in another way. The depression of the zero point of a thermometer one whole degree centigrade following the heating of the instrument to the boiling point may be considered a large depression. The fatigue dilatation is in this case 1 per cent of the apparent expansion of the mercury between 0° and 100°. For this range of temperature the coefficient of cubical expansion of mercury is 0.000182 per degree, or 0.0182 for 100°. If the coefficient of cubical expansion of glass be taken at 0.000028, the expansion of the bulb of the thermometer on being heated from 0° to 100° would be 0.0028 of its capacity at 0°. The apparent expansion of the mercury contained in it would therefore be 0.0154 of its volume, 1 per cent of which is 0.000154 of the volume of the mercury; i. e., the fatigue dilatation would be 5.5 per cent of the total expansion of the glass. If a liter flask be heated from 20° to the boiling point of water, its capacity is increased by expansion 2.24 cc. The fatigue dilatation, taken as 5.5 per cent of the total expansion, would then be 0.123 cc; i. e., the capacity of the flask would be 0.123 cc greater immediately after cooling than it was before it was heated. Even an error as great as this would be negligible in ordinary routine analytical work, but the experiments cited above and the amount of depression of the zero

point exhibited by thermometers both indicate that the fatigue dilatation of laboratory glassware of ordinary quality is much less than 5.5 per cent of the total expansion it undergoes when heated. It is therefore evident that the hypercriticisms referred to above have no practical value.

I have recently completed a series of check standardizations of three flasks by the two official methods. The results are set forth in the following table:

Check standardizations of three flasks.

Date.	Temperature.	Barometer reading.	Flask No. 17.		Flask No. 77.		Flask No. 79.	
			Weight of water at T°.	Calculated weight of water at 100°.	Weight of water at T°.	Calculated weight of water at 100°.	Weight of water at T°.	Calculated weight of water at 100°.
1900.	° C.	Mms.	Grams.	Grams.	Grams.	Grams.	Grams.	Grams.
November 12.....	21.0		27.013	26.0042	51.455	49.5334	48.1915	46.3917
Do	22.7		27.002	26.0023	51.440	49.5355	48.175	46.3914
Do	29.3		26.959	26.0019	51.358	49.5337	48.098	46.3905
November 13.....	36.2		26.903	26.0012	51.253	49.5350	47.998	46.3891
Means.....				26.0024		49.5344		46.3907
Maximum variations from mean..				0.0018		0.0011		0.0016
Maximum variations of two results.				.0030		.0021		.0026
	(Corrected.)							
November 14.....	¹ 99.95	765	26.008		49.540		46.397	
Do	¹ 100	764.5			49.547		46.388	
Do	100	764			49.550		46.407	
November 15.....	100	766.8			49.540		² 46.355	
Means.....					49.5443		46.3973	
Maximum variations from means					0.0057		0.0097	
Maximum variations of two results.					.0100		.0190	
Variation of means from mean calculated results					+.0099		+.0066	
November 15.....	21.65		27.010	26.0046	51.453	49.5377	48.188	46.3942

¹ These temperatures are given as accurately as they could be read with an accurate thermometer. The correction for exposed column of mercury varied from 0.8 to 0.9° C. A verified short thermometer was not available.

² Omitted in the calculation of averages.

RECOMMENDATIONS.

In view of these experiments and the facts set forth in the above discussion, I beg to recommend that the referee on dairy products for next year be instructed to consider the following changes in the methods for determining the specific gravity of fats and oils and present them at the next meeting for adoption by the association, together with such modifications and additions as may be found advisable in the meantime:

(1) That this determination be not made "at the temperature of boiling water," but at 100° C., using, to attain this temperature, a bath of glycerol or some other suitable substance soluble in water or miscible therewith; that the bath be so arranged that the controlling thermometer can be immersed to the 100° mark.

(2) That the ordinary specific-gravity flask with perforated stopper be replaced by the Sprengel tube or a modification thereof.

(3) That the flask or tube have a capacity of not less than 100 cc when the amount of the sample and the capacity of the balance will permit; in other cases a vessel of 50 cc capacity may be used.

(4) That the standardization of flasks by calculation be limited to those made of glass, of which the coefficient of cubical expansion is known with precision to the sixth decimal place.

Further, I recommend that this association do now make the following change in the method for standardizing flasks for the determination of the specific gravity of fats and oils:

Bulletin No. 46. revised edition, page 51, under "Optional method of standardizing flasks," insert the following in place of the method there given:

The following formula may be used for calculating the weight of water (W^{T^0}) which a given flask will hold at T^0 (weighed in air with brass weights at the temperature of the room) from the weight of water (W^t) (weighed in air with brass weights at the temperature of the room) contained therein at t^0 :

$$W^{T^0} = W^t \frac{d^{T^0}}{d^t} [1 + \gamma (T - t)]$$

d^T = the density of water at T^0 .

d^t = the density of water at t^0 .

γ = the coefficient of cubical expansion of glass.

SATURDAY—EVENING SESSION.

The meeting was called to order by the president at 7 o'clock.

The report of the special committee on the subdivision of food work was presented as follows:

REPORT OF THE SPECIAL COMMITTEE ON THE SUBDIVISION OF FOOD WORK.

Resolved, That it be made the duty of the referee on liquor and food adulteration to associate with him such members of the association as he may see fit, each of whom shall be instructed to investigate and report on one or more of the following subjects:

- (1) Meat and fish.
- (2) Fats and oils (not including dairy products or their substitutes).
- (3) Cereal products.
- (4) Infants' and invalids' foods.
- (5) Saccharine products (including confectionery).
- (6) Vegetables (canned, dried, or otherwise preserved).
- (7) Tea, coffee, and cocoa.
- (8) Spices and condiments.
- (9) Vinegar.
- (10) Flavoring extracts.
- (11) Fruit products.
- (12) Fermented and distilled liquors.
- (13) Baking powder and baking chemicals.
- (14) Preservatives.
- (15) Dyes.

Adopted.

The president next called for the report of the committee on recommendations.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

ASH.

(1) The committee recommends the use of calcium acetate and burning ash in some way to prevent volatilization.

Adopted.

CATTLE FEEDS.

(1) Phloroglucin method (Bul. 46, rev. ed., p. 25):

(a) The 5th line be changed so as to read: "regulated so as to distill 30 cc in about ten minutes, the distillate passing through a small filter paper."

Adopted.

(b) In the 6th line, after "dilute acid," the following words be added: "added in such a manner as to wash down the particles adhering to the sides of the flask."

Adopted.

(c) The distillation be continued until the distillate amounts to 360 cc.

Adopted.

(d) In the 8th line the words "free from diresorcol" be changed to "purified if necessary."

Adopted.

(e) In the 14th line the words "over night" be changed to "at least twenty hours."

Referred to referee for ensuing year.

(f) The method of purifying the phloroglucol devised by Mr. Fraps¹ be adopted and added to the paragraph entitled, "Qualitative test of the purity of the phloroglucol." (Bul. 46, p. 26.)

Adopted.

(2) Galactan method (Bul. 46, p. 26): It is recommended that in the fourteenth line, after "ammonia," the following be added: "The filter paper and contents are then washed several times with hot water by decantation, the washings being passed through a filterpaper, to which the material is finally transferred and thoroughly washed."

Adopted.

(3) It is finally recommended that the terms "phloroglucol," "di-resorcol," and "furfural" be adopted.

Adopted.

CHANGES IN BULLETIN 46, REVISED.

(1) Page 47, in paragraph 2 (a), "soluble acids," insert in the third line the following sentence: "Or the saponification may be conducted in a flask connected with a reflux condenser."

Adopted.

(2) On page 52, sixth line from bottom, add the sentence: "These discs should be prepared a day, or at least a few hours, before using."

Adopted.

¹See p. 112.

It was moved and adopted that the question of the number of hours the discs should be made before using be referred to the referee for the ensuing year.

(3) On page 48, eighth line from bottom, change the words "between 1 and 2 grams" to "approximately 2.5 grams." At the end of the same sentence, insert (after a semicolon) the words "or the saponification may be conducted in a flask with a reflux condenser."

Adopted.

It was moved and adopted that the secretary be requested to insert in the next edition of the methods the latest edition of the revision of the atomic weights of the committee on atomic weights of the American Chemical Society.

By vote of the association, the editor was authorized to change the quantities of sodium thiosulphate (p. 50, Bulletin 46) to meet new atomic weights.

DAIRY PRODUCTS.

(1) The committee recommends that paragraph 3, p. 55 of Bulletin 46, entitled "Official optional method for determining casein in milk," be changed to read as follows:

3. *Official optional method for determining casein*—To 10 cc of milk add 50 cc of distilled water at 40°, then add 2 cc of alum solution saturated at 40° or higher. Allow precipitate to settle, transfer to a filter, and wash. The washed precipitate and filter paper are digested as in regular Kjeldahl method, as outlined in the official method, paragraph 1. Calculate casein by multiplying the amount of nitrogen found by 6.25.

Adopted.

(2) The committee recommends that paragraph 4, page 55, of Bulletin 46, entitled "provisional optional method for determining albumin in milk," etc., be changed to read as follows:

Provisional optional method for determining albumin in milk—To the filtrate from the preceding add 10 cc of 20 per cent tannin solution, heat, filter, wash with distilled water, and determine nitrogen by the Kjeldahl method. Multiply the result by 6.25 for albumin.

This recommendation went over without action.

The recommendations made by Mr. Ewell¹ relative to the determination of the specific gravity of fats and oils, as well as the change recommended under the "Optional method of standardizing flasks," Bulletin 46, revised edition, p. 51, were favorably reported and adopted.

Mr. Carr then presented the report on tannin.

REPORT ON TANNIN.

By OMA CARR, *Referee*.

Owing to disappointment in not receiving a hide-powder suitable for the official method, the referee delayed sending out the samples for cooperative work until November 1. On that date samples of liquid oakwood extract and solid quebracho

¹ See p. 125.

extract, selected as representing typical commercial materials, were sent to the collaborators. Reports were received from eight analysts, giving the results to be discussed in the following pages. Work done under the referee's direction will be entered first, to be followed by a tabulation of the collaborative analyses.

SOLUBLE SOLIDS.

In nearly all tanning materials the anhydrides, gums, phlobaphenes, etc., grouped as insolubles, are present in the analysis solution in such state of fine division that filtration, even through finest paper, will not effect complete removal. In such cases the method permits the use of kaolin, previously washed in a portion of the solution, with the view of "filling" the paper and thus obstructing the passage of the insoluble matter. While this procedure accomplishes the object of a clear filtrate, it is open to the objection that kaolin is of varying composition and that the increased time of contact with the paper increases the absorption and lowers the result as to soluble solids.

With the aim of overcoming the paper absorption, the referee tried various methods of mordanting the paper and coloring with a dye without much success beyond pointing the way for further experiment. The paper was saturated with a solution of gallotannic acid and struck with a basic dye. Looseness of attraction prevented a good result, and subsequent washing removed a large amount of color. Paper so prepared, however, possessed less absorption power than the untreated fiber and the filtering power remained good. Possibly further experiment may enable the paper makers to produce an absorption-free fiber. As the fiber of the paper acts basically in tannin attraction, it was thought that the addition of a volatile acid, inert as to chemical action on the constituents of the solution, might overcome the attraction. Experiments were conducted on a solution of oakwood extract, adding 0.36 gm of acetic acid, in weak solution, to each 100 cc before filtration, and passing through plain and pleated No. 590, 15 cm paper.

Effect of the addition of acetic acid.

Volume of filtered solution taken for determination.	Soluble solids.	
	Plain paper.	Pleated paper.
	<i>Per cent.</i>	<i>Per cent.</i>
First 50 cc.....	41.01	41.90
Second 50 cc	41.17	41.95
Third 50 cc.....	41.66	42.27

It will be noted that the rate of filtration was higher with the pleated paper.

The action of plain folded paper, without acid, is shown in the following table, and it will be seen that there was a decrease rather than an increase in the rate of filtration:

Soluble solids obtained without the addition of acetic acid and the use of plain folded paper.

Method of filtration and volume of filtered solution taken.	Soluble solids.
	<i>Per cent.</i>
75 cc through, 50 cc taken.....	41.07
75 cc through same paper, 50 cc.....	40.95
75 cc through same paper, 50 cc.....	40.79

The action of kaolin and barytes, with the same solution, and no acid gave the following results:

Comparison of effect of kaolin and barytes on the amount of soluble solids obtained.

Method of filtration.	Soluble solids.
	<i>Per cent.</i>
150 cc passed, then 5 gm kaolin, washed in solution.....	39.52
150 cc passed, then 10 gm kaolin, washed in solution.....	37.34
150 cc passed, then 5 gm barytes, washed in solution	40.79
150 cc passed, then 10 gm barytes, washed in solution	40.49

The use of a 100 cc solution, 0.02 gm of lead acetate, 0.36 gm of acetic acid, correcting for removal by lead and dilution, and filtering through No. 590, 15 cm, pleated paper, gave triplicate determinations, as follows: 41.92, 41.70, 41.85 per cent soluble solids. The filtrates were brilliantly clear.

Work was undertaken along the same lines with solid quebracho extract, with the following results:

Comparison of effect of kaolin and barytes on the amount of soluble solids obtained, using plain folded paper.

Method of filtration.	Soluble solids.
	<i>Per cent.</i>
150 cc passed, then 5 gm kaolin, washed in solution.....	79.82
150 cc passed, then 10 gm kaolin, washed in solution.....	74.67
150 cc passed, then 5 gm barytes, washed in solution	76.36
150 cc passed, then 10 gm barytes, washed in solution	76.80

Effect of the addition of acetic acid, using pleated paper.

Volume of filtered solution taken for determination.	Soluble solids.
	<i>Per cent.</i>
First 50 cc.....	77.05
Second 50 cc.....	76.82
Third 50 cc.....	76.71

The filtrates were clear.

By the use of 100 cc of solution, 0.02 grams lead acetate, filtered through asbestos, and corrected for lead removal and dilution, triplicate determinations were obtained as follows: 74.41, 74.38, 74.40 per cent soluble solids. The filtrates were brilliantly clear.

The use of 100 cc solution, 0.02 gm acetate lead, 0.32 gm acetic acid, filtered through No. 590, 15 cm, pleated, and corrected for removal by lead and dilution gave the following triplicate determinations: 74.95, 75.24, 75.15 soluble solids. The filtrates were brilliantly clear.

A review of these results develops the following conclusions:

- (1) Filtration through paper, plain or pleated, without "assistants" does not totally remove insolubles.
- (2) Absorption is largely a function of the time the solution is in contact with the paper, being less for free filtering and greater for viscous solutions.
- (3) Acetic acid modifies the basic nature of the paper.

(4) It is possible to secure concordant results by the use of lead acetate, acetic acid, and paper.

(5) As the method stands for soluble solids, it gives low figures for the oakwood and high for the quebracho, and this will hold good in the comparison of any materials so differing.

The referee is of the opinion that results reported to this association in the past, and the experience of the International Association of Leather Trades Chemists, are ample support for the retention of Schleicher and Schüll, No. 590, 15 cm, for all filtrations. It will be found, however, in substituting the pleated for the simple folded form now stipulated, that the passage of the liquor is expedited and the absorption is reduced.

Finally, with reference to the subject of soluble solids, the referee would suggest the following optional method and the substitution of "barytes" for "kaolin" in the official method:

To 100 cc of the solution add 10 cc of a solution of acetate of lead containing 4 grams of the salt per liter, adding the reagent drop by drop from a burette and stirring meanwhile. Now add 10 cc of a solution of acetic acid containing 36 grams glacial acid per liter, stirring. Throw on double-pleated filter, reject until clear, evaporate 50 cc, and dry.

To another portion of 100 cc repeat the foregoing, except that 20 cc of lead acetate solution shall be used.

Residue from first treatment shall be multiplied by 1.2, and that from the second by 1.3, to bring back to the original 100 cc of solution. Add to the corrected first residue the difference between corrected first and second residues, and calculate as soluble solids. This corrects for the dilution and removal by the lead salt.

HIDE POWDER.

Events of the past summer have emphasized the necessity that hide powder possess the qualifications specified by the method. The principal importer of Vienna powder has supplied the referee with samples from four different lots of powder, none of which approach the standard of insolubility and absorption power. The Vienna powder, generally used because of its close adherence to the limits of insolubility and absorption, has recently shown such wide departure as to fall wholly without the range of allowable variation.

Inasmuch as the determination of tannin is dependent upon the character of the powder employed, no analysis can be truthfully reported as by the official method unless the powder employed shall fall within the prescribed limits. This distinction between powders within and without these limits is not only essential but vital to the value of results, from either the scientific or commercial standpoint. Many of the largest consumers of tanning materials base heavy contracts as to quality upon the official method, and unless it shall be closely stipulated that the powder employed shall answer the requirements of the method, something worse than controversy may result.

The referee, therefore, recommends that to Section VIII, page 80, shall be added the following paragraph:

"(c) Any analysis made with a powder which does not fulfil the conditions of the preceding paragraphs, shall not be reported as by this method."

The referee has made several experiments with powders falling without the requirements of the method, with results emphasizing the necessity for the foregoing recommendation.

ACID HIDE POWDERS.

The four powders considered in the following table were markedly acid, and when suspended in water preparatory to an analysis softened into a jelly-like mass which resisted the drying action of squeezing in linen. In order to remove the acidity of

the powder it was necessary to use considerable quantities of sodium carbonate in the washing water:

Determination of nontannins using acid hide powders.

Methods.	Nontannins.
	<i>Per cent.</i>
Powder I:	
Without carbonate	16.93
With carbonate, washed three times.....	16.34
With carbonate, official.....	16.23
Powder II:	
Official.....	17.89
With carbonate, official	17.05
Powder III:	
Without carbonate, official	18.31
With carbonate, official.....	17.12
Powder IV:	
Without carbonate, official.....	17.68
With carbonate, official.....	16.74

When it is necessary to resort to an expedient, such as delicate neutralization, it is likely that different operators would return widely ranging results.

WASHING THE POWDER.

A good powder was used and washed with the following results:

Effect of variations in washing on the amount of nontannins obtained.

Number of washings.	Nontannins.
	<i>Per cent.</i>
Not washed.....	19.74
Washed once in shaker.....	17.90
Washed twice in shaker.....	18.04
Washed three times in shaker.....	17.96
Washed four times in shaker.....	17.98

VARYING QUANTITIES OF HIDE POWDER.

A large quantity of powder was washed and pressed and duplicate determinations made with increasing quantities of wet pressed hide, which contained 75 per cent of moisture:

Determination of nontannins with increasing quantities of hide powder.

Amount of hide powder.	Nontannins.	Mean.
	<i>Per cent.</i>	
50 grams wet hide.....	{ 17.33 17.34 }	17.34
60 grams wet hide.....	{ 16.61 16.63 }	16.62
70 grams wet hide.....	{ 16.11 15.96 }	16.04

Inasmuch as the moisture content of the wet pressed hide is variable with the physical condition thereof, a definite statement of the dry hide present in the wet

pressed cake must be made. Results are concordant for any definite quantity, but the fineness of some powders permits great loss in squeezing, and the actual dry hide present in the solution thereby becomes variable. The referee thinks it wise to stipulate that the dry hide present in the wet cake taken for the determination shall fall between 12 and 13 grams.

VOLUME OF SOLUTIONS FOR DRYING.

Owing to the sensitiveness of tanning materials to the heat employed in drying, it is believed that the use of volumes yielding residues of from 400 to 500 milligrams is preferable to volumes giving greater residues. It is believed that if, instead of 100 cc 50 cc shall be used for total and soluble solids the resultant residue may be dried to constant weight in less time and at less risk of decomposition or oxidation. The experience of the referee is that residues of 400 milligrams may be dried to nearly constant weight on the steam or water bath in five hours, passing through an air or water oven for a half hour to remove surface moisture. While some laboratories are equipped to use volumes of 100 cc, others are not and it is believed that the smaller volume and residue will be advantageous in bringing the entire determination within one working day.

The referee would recommend, therefore, the substitution of 50 cc wherever in the method 100 cc is stipulated for drying.

Summary of analyses.

OAKWOOD EXTRACT.

Analyst.	Total solids.	Soluble solids.	Insoluble.	Tannin.	Nontannin.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	43.45	42.24	1.21	25.64	16.60
2.....	43.37	42.54	.83	25.78	16.76
3.....	42.74	42.13	.61	25.20	16.93
4.....	42.53	40.65	1.88	24.72	15.93
5.....	42.85	42.22	.63	24.07	18.15
6.....	43.23	42.53	.70	25.07	17.46
7.....	42.94	42.29	.65	25.25	17.01
8.....	43.29	42.26	1.03	25.11	17.15
Averages	43.05	42.11	.94	25.11	16.99
Maxima	43.45	42.54	1.88	25.78	18.15
Minima	42.53	40.65	.61	24.07	15.93

QUEBRACHO EXTRACT.

1.....	87.33	74.00	13.33	64.70	9.30
2.....	86.71	74.52	12.19	65.56	8.96
3.....	87.08	76.33	10.75	66.84	9.49
4.....	90.13	79.82	10.31		
5.....	87.45	75.80	11.65	67.08	8.72
6.....	87.40	76.29	11.11	68.35	7.94
7.....	85.98	75.10	10.88	67.47	7.63
8.....	86.45	74.45	12.00	66.25	8.20
Averages	87.32	75.70	11.59	66.61	8.61
Maxima	90.13	79.82	13.33	67.47	9.49
Minima	85.98	74.00	10.31	64.70	7.63

The above eight analyses exhibit some improvement over the results previously obtained by collaboration. The quebracho is a severe trial on the accuracy of

the method, owing to the high tannin and the extreme fineness of the insolubles in the analysis solution. Variations in nontannins may be largely attributed to variations in character of hide powder used, as the analysts used the powder employed in their respective laboratories. Given a soluble solids determination accurate to 1 per cent on a solid extract, it is likely that the principles of the nontannin determination as now laid down in the method may be considered satisfactory. The range from maximum to minimum is relatively greater for all determinations, except nontannins, in the oakwood than in the quebracho.

THE METHOD.

The following modifications of the official method, Bulletin 46, revised, page 79, VIII, Methods for the Analysis of Tanning Materials, are recommended:

III. MOISTURE. (b) 2. Read "For eight hours at 100° to 110° in an air bath."

IV. TOTAL SOLIDS. For "100 cc" read "50 cc."

V. SOLUBLE SOLIDS. For "double-folded filter" read "double-pleated filter." For "100 cc" read "50 cc." For "kaolin" read "barytes."

Add paragraph giving optional method, as follows:

Optional method.

(a) To 100 cc of the solution add 10 cc of a solution of lead acetate (4 grams per liter), adding the reagent drop by drop from a burette and stirring meanwhile. Add 10 cc of a solution of acetic acid (36 grams glacial acid per liter), stirring. Throw on double-pleated filter, reject until clear, and evaporate and dry 50 cc.

(b) On another portion of 100 cc repeat the foregoing, except that 20 cc of lead acetate solution shall be used.

Residue from (a) shall be multiplied by 1.2, and from (b) by 1.3, to bring back to the original 100 cc. Add to the corrected weight of the residue from (a) the difference between (a) and (b) and calculate the found residue to soluble solids. This corrects for dilution and removal by lead.

VI. NONTANNINS. For lines 6-9 substitute the following: "Weigh the remaining three-quarters, which must contain between 12 and 13 grams of dry hide; add to 200 cc of the solution and shake ten minutes. Throw on funnel with cotton plug in stem, return until clear, evaporate 50 cc, and dry."

VIII. TESTING HIDE POWDER. (a) For "100 cc" read "50 cc," and for "10 mg" read "5 mg." (b) For "100 cc" read "50 cc."

Add paragraph (c), as follows:

(c) Any analysis made with a powder which does not fulfill the conditions of the preceding paragraphs shall not be reported as by this method.

At this stage of the proceedings, Mr. Krug presented the following papers:

REPORT ON COMPARATIVE HIDE-POWDER TESTS.

By WILLIAM H. KRUG.

During the last year various chemists engaged in the analysis of tanning materials have frequently complained of the character of the hide powder they were using, and have ascribed unsatisfactory results to variations in its absorbent power. The hide powder generally used in this country is made by the Vienna Experiment Station, and has, until recently, given almost universal satisfaction. Within the last year, however, it has undergone marked changes in that it is quite acid in reaction and retains a large amount of moisture after being squeezed even when repeatedly washed with water. When neutralized with sodium carbonate it loses this power of retaining water, but also decreases in its power of absorption with reference to tannin. It is generally claimed that this acid hide powder when used without previous neutralization gives a figure for nontannins which is too low, and thus in turn results in too high a tanning value being ascribed to the material under examination.

It being evident that this matter is of great importance to all chemists interested in this branch of analytical chemistry, I some time ago sent a sample of quebracho

extract, which had been ground and carefully mixed so as to render it strictly uniform in composition, to Mr. Alsop, of the United States Leather Company, and Mr. Teas, of the Elk Tanning Company. The sample was accompanied by a sufficient quantity of Vienna hide powder to make several determinations, and I suggested that the extract be analyzed both with and without previous neutralization of the powder with sodium carbonate. Unfortunately Mr. Teas was unable, through pressure of other work, to make more than one analysis in which he used the hide powder on hand at his laboratory. As this was derived from the same source as that sent by me for the comparative work, the two powders are probably identical. Mr. Alsop sent quite a complete report embodying results obtained both with hide powder in use at his laboratory and that sent him. The results are contained in the following table:

Comparative analyses of quebracho extract both with and without previous neutralization of hide powder.

No.	Analyst.	Method employed.	Total sol-ids.	Soluble sol-ids.	Reds.	Non-tannins.	Tannins.	Remarks.
			<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	
1	Alsop...	Laboratory hide, official method, not neutralized.	85.68	76.74	8.94	10.25	66.49	Showed traces of tannin in nontannin filtrate. Filtrate was clear.
2	do...	Laboratory hide, official method, neutralized.	85.68	76.74	8.94	12.74	64.00	Filtrate was turbid.
3	do...	Laboratory hide, official method, treated with one-third the amount of Na_2CO_3 necessary to neutralize.	85.68	76.74	8.94	9.78	66.96	Filtrate was clear and contained no tannin.
4	do...	Hide powder sent for comparative test, not neutralized.	85.68	76.74	8.94	10.68	66.06	Hide powder washed according to official method.
5	do...	Hide powder sent for comparative test, treated with one-third the amount of Na_2CO_3 necessary to neutralize.	85.68	76.74	8.94	10.79	65.95	Hide powder washed according to official method.
6	do...	Laboratory hide, not neutralized.	85.68	76.74	8.94	10.45	66.29	In these analyses the hide powder was washed according to the method adopted in Mr. Alsop's laboratory.
7	do...	Laboratory hide, treated with one-third the amount of Na_2CO_3 necessary to neutralize.	85.68	76.74	8.94	9.90	66.84	
8	do...	Laboratory hide, neutralized.	85.68	76.74	8.94	12.14	64.60	
9	Teas....	Laboratory hide.	84.95	75.05	9.90	10.65	64.40	Treatment of hide powder not stated.
10	Krug...	Hide powder sent for comparative test, not neutralized.	85.35	77.43	7.92	10.08	67.35	20 grams hide powder used, washed according to official method.
11	do...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	11.74	65.69	20 grams hide powder used, washed in a beaker ten times by decantation, using a liter of water each time. Nontannin filtrate showed traces of tannin and was turbid.

Comparative analyses of quebracho extract both with and without previous neutralization of hide powder—Continued.

No.	Analyst.	Method employed.	Total sol-ids.	Soluble sol-ids.	Reds.	Nontannins.	Tannins.	Remarks.
			<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	
12	Krug ...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	10.49	66.94	30 grams of hide powder used, washed in a beaker ten times by decantation, using a liter of water each time. Nontannin filtrate showed traces of tannin and was turbid.
13	do ...	Hide powder sent for comparative test, neutralized.	85.35	77.43	7.92	9.81	67.62	40 grams of hide powder used, washed in a beaker ten times by decantation, using a liter of water each time. Nontannin filtrate was clear and showed no tannin.

Mr. Alsop describes the method of washing the hide powder in use in his laboratory as follows:

Enough hide to make the determination is treated with about one hundred times its weight of water. If too acid to squeeze well, one-third the amount of sodium carbonate necessary to neutralize is added. It is then squeezed by hand through linen, treated with the same amount of water, and squeezed once more. If sodium carbonate has been added this washing is repeated again. It is then squeezed in a press and 50 grams taken for nontannins. A portion is dried for determination of moisture. I can obtain more uniform results by this method than by the official method where 20 grams are taken for each determination.

A comparison of the results obtained by Mr. Alsop and myself shows a close concordance with reference to the nontannins when the hide powder was not neutralized, and this is true both of the hide powder sent him for the comparative work and that in use in his own laboratory. When the acidity of the hide powder is neutralized, however, its absorption power becomes less, the nontannin filtrates are turbid, and show traces of tannin. It was found that after neutralization it requires at least 40 grams of hide powder to obtain a filtrate free from tannin. The use of such a quantity is out of the question on account of the cost of the material.

The results clearly show the influence which the acidity of the hide powder exercises on the percentage of nontannins obtained and render it evident that something should be done to enable chemists to obtain a neutral powder. While Mr. Alsop's method of treating with one-third the amount of sodium carbonate necessary for neutralization may be good, the question nevertheless arises if this is not merely choosing a mean between two evils. The accuracy of the official method depends to a very great extent on the character of the hide powder used, and every precaution should be employed to avoid sources of error in this feature of the method. I would therefore suggest that the matter be taken up by the association, possibly by inserting a clause in the present method, directing that the hide powder used in the determination of the nontannins shall be neutral. The method as it now stands specifies certain standards as to the maximum amount of soluble matter which shall be present and the absorption power. It appears, however, that these restrictions do not cover fully the exigencies of the case.

The following paper was then presented:

A COMPARISON OF THE INTERNATIONAL FILTER-TUBE METHOD AND THE OFFICIAL HIDE-POWDER METHOD.

By H. W. WILEY and WILLIAM H. KRUG.

The filter-tube method has been employed for many years by chemists on the continent in the analysis of tanning materials. It was officially adopted by the International Association of Leather Trades Chemists at a conference held in England in 1897, and has since been retained by that association. The objection made by American chemists to this method is that it yields a figure for nontannins which is too low, and thus gives the tanning material a higher value than it should have. At the last meeting of the international association, which was held in Paris last summer, and which one of us attended as American delegate, it was arranged with Professor Procter to make a series of comparative tests on tanning materials which were to be furnished by him. These materials comprised a sample each of oak-wood extract, chestnut-wood extract, liquid quebracho extract, mimosa extract, solid quebracho extract, sumac, valonia, golden wattle, and mimosa bark. Upon arrival of the samples it was found that the bottle containing the liquid quebracho extract was broken, and the analyses of the sumac and golden wattle were interrupted by the breaking of the bottles during extraction. The analyses by the filter-tube method were made in Professor Procter's laboratory, while those by the official method were made in the Division of Chemistry, United States Department of Agriculture. The results obtained are compiled in the following tables:

Comparison of results obtained by the official and the filter-tube methods in the analysis of tanning materials.

Constituents.	Oak-wood extract.		Chestnut-wood extract.		Mimosa extract.		Solid quebracho extract.	
	Filter-tube method.	Official method.	Filter-tube method.	Official method.	Filter-tube method.	Official method.	Filter-tube method.	Official method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture	59.60	59.50	59.80	60.36	50.80	50.28		16.84
Total solids by dilution		40.34		39.74		49.90	81.10	83.39
Soluble solids by dilution		40.02		38.89		49.70	69.10	75.20
Reds	0.40	0.32	0.80	0.85	0.00	0.20	12.00	8.19
Nontannins	13.00	14.66	10.60	13.11	9.00	12.33		14.01
Available tannins ..	27.00	25.36	28.80	25.78	40.20	37.37		61.19

Constituents.	Valonia.		Mimosa bark.	
	Filter-tube method.	Official method.	Filter-tube method.	Official method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture	12.20	12.60	10.80	
Total solids in extract ¹		53.51		47.04
Soluble solids in extract ¹		52.69		43.30
Reds in extract ¹		0.82		3.74
Nontannins	12.90	13.20	10.70	10.11
Available tannins	40.30	39.49	32.30	33.19
Total insoluble matter	34.60	34.71	46.20	

¹Calculated to original material.

The results show in general a very good agreement as to the amount of moisture present in the samples. The same is true of the reds, except with the solid quebracho extract, where the high percentage obtained with the filter-tube method shows the effect of rapid cooling of the diluted extract, as specified by this method, upon the sensitive tannin which it contains. The chief point of interest in these figures is naturally the nontannins, as these indicate the variation caused by the different application of the hide powder. In every case except one, that being mimosa bark, the filter-tube method gives lower nontannins than the official method and correspondingly higher tannins. It is evident, therefore, that a true international method is an absolute necessity. Large quantities of tanning materials are annually sold to American importers, the European merchant demanding payment on the basis of the results obtained by the filter-tube method. The American importer in turn is forced to sell to the tanner on the basis of figures obtained by the official method, the result being constant friction between buyer and seller. It gives us pleasure to state that the first step toward a uniform method has been taken, the International Association at its last conference in Paris having adopted the method of cooling the diluted extracts used in this country. It is to be hoped that the efforts in this direction will be continued, and will result finally in the development and universal adoption of a truly international method for the analysis of tanning materials.

Mr. Sawyer gave a very interesting talk on various debatable points in the present method for the analysis of tanning materials. He advocated the use of a smaller volume of the solutions in determining total and soluble solids, and suggested a series of comparative experiments on various methods of filtration.

Mr. WILEY. I attended the International Congress of Leather Chemists, which met in Paris in August, 1900. The president of this congress, Prof. H. R. Proctor, is well known to chemists in this country, especially those engaged in the tanning industries.

I was most cordially received as the representative of the Association of Official Agricultural Chemists and invited to a seat of honor at the president's right, and asked to take part in the discussion. Finding that there was no American representative in the congress, I decided to become a member thereof, in order that we might be not only officially but actively represented. The proceedings of the congress were conducted in three languages, which made the sessions rather monotonous. Each member of the congress spoke in his native language, and his speech was afterwards repeated in the other two languages of the congress, French, German, and English being the three languages recognized.

In adopting methods of research and analysis the processes recommended by the Association of Official Agricultural Chemists received due consideration. In fact, an earnest effort was made to harmonize the methods employed on the Continent with those employed by our own chemists. Some of our methods were adopted en bloc and others in a more or less modified form. It is evident that with a very little effort we may be able to unify the methods in vogue both in America and in Europe. This would be extremely useful, not only from a scientific point of view, but especially from the commercial aspect, since

in this case analyses made by chemists on the two continents would be conducted by the same methods and be strictly comparable.

The banquet tendered the congress by the syndicate of leather merchants of Paris was an elaborate affair given at the Hotel Continental, and was one of the very pleasing features of the meeting.

Mr. Wiley then spoke on the question of the unification of methods of fertilizer analysis as follows:

The international committee on the unification of methods of fertilizer analysis presented its report to the Fourth International Congress of Applied Chemistry, which met in Paris in July, 1900. The president of this commission was Dr. Ritter von Gruber, and I had the honor to be the American member.

A basis of international agreement for fertilizer analysis was adopted by the committee, in which all the leading principles were recognized as standard and the details of manipulation were left open for further consideration. The committee considered that it was a wiser plan at this period to agree upon the essential principles, and to cite the standard authorities of the United States, Germany, and France for the details at the present time. The report was printed in three languages, German, French, and English, stating the essential points of agreement and citing the standard authorities of different countries. This report of the committee was presented to the section of agricultural chemistry, and, after a prolonged discussion, it was unanimously adopted.

This is a matter for congratulation on the part of the Association of Official Agricultural Chemists, because it was a recognition by this international congress of the valuable work which has been accomplished by this body, and the official methods of analysis are now for the first time cited in an authoritative publication in Europe for the guidance of analysts throughout the world.

The report of the committee on recommendations was now called for.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

TANNIN.

The committee recommends the adoption of the amended method for tannin, as given by Mr. Carr in his report. (See p. 133.)

Adopted.

Mr. Sawyer suggested that the referee for the ensuing year investigate the evaporation of smaller volumes of solution than 50 cc and weighing of smaller residues than 0.4000 gram.

Adopted.

Mr. Sawyer also moved that the referee for the ensuing year study the process of sedimentation and of centrifuging, to replace filtration.

Adopted.

Mr. Huston suggested that, in connection with the above study, the referee try filtering through a Pasteur filter instead of paper.

Adopted.

The referee on insecticides being absent, the report was read by the secretary, as follows:

REPORT ON FUNGICIDES AND INSECTICIDES.

By LOUIS A. VOORHEES, *Referee.*

In presenting the following report your referee begs to say a word of explanation and apology. Having been continued as associate, he awaited the pleasure of the referee, but heard nothing until suddenly informed that the latter, for excellent reasons, would be unable to conduct the work, which therefore would devolve upon the writer. This occurred in the spring, when the large amount of work in the fertilizer control was beginning to require the entire attention for the summer of the chemical force at the New Jersey experiment station. This state of affairs was submitted to the president of the association, and it was accordingly agreed that since it would be impossible to present any analytical work, the report should consist of a further compilation of methods.

A year ago the writer, in his report as associate referee, submitted methods for the examination of fungicides and insecticides, which were adopted as provisional methods by the association. The methods were selected entirely by the writer. This year it was deemed advisable to get the opinion of the station workers, and a circular letter was sent to all the experiment stations and to several commercial laboratories, as follows:

DEAR SIR: The undersigned, owing to the resignation of Dr. de Schweinitz as referee of the Association of Official Agricultural Chemists on fungicides and insecticides, has been appointed by the president to undertake the work.

Last year, as associate referee, and in the absence of Dr. de Schweinitz in Europe, the undersigned proposed certain methods for the determination of the essential constituents of the various fungicides and insecticides, which methods were adopted provisionally by the association, and published in the annual proceedings for 1899, page 101.

Will you kindly advise the writer if you have employed any of these methods in your laboratory at any time, and with what success? Also please point out any difficulties met with or modifications proposed, or give a detailed statement of your own methods for these determinations. The results of any analytical work, comparative or otherwise, will be appreciated, and due credit given in every instance.

The determinations involved are:

Alkalinity in potash and soda lyes.

Arsenic in arsenicals.

Copper in copper carbonate.

Cyanogen in potassium cyanid.

Formaldehyde in formalin.

Nicotin in tobacco.

Hoping to receive an early reply, I remain,

Yours very truly,

LOUIS A. VOORHEES,
*Referee Association of Official Agricultural Chemists
on Fungicides and Insecticides.*

Twenty-seven replies were received, 14 stating that no work has been done along these lines, and the remaining 13 communicating more or less extensive results of work and experience. The latter were received from the following-named persons:

E. E. Ewell	U. S. Department of Agriculture, Washington, D. C.
E. W. Hilgard	California Experiment Station.
A. L. Winton	Connecticut Experiment Station.
S. Avery	Idaho Experiment Station.
A. M. Peter	Kentucky Experiment Station.
H. J. Patterson	Maryland Experiment Station.
C. A. Goessman	Massachusetts Experiment Station.
N. E. Wilson	Nevada Experiment Station.
M. B. Hardin	South Carolina Experiment Station.
C. H. Jones	Vermont Experiment Station.
R. J. Davidson	Virginia Experiment Station.
Elton Fulmer	Washington Experiment Station.
Wm. S. Myers	Rutgers College.

No methods were communicated for the determination of alkalinity in lyes or soaps or of cyanogen in potassium cyanid. Mr. R. J. Davidson used Kissling's method for nicotin, while Mr. A. M. Peter, on the other hand, writes as follows:

Recently I have made a determination of nicotin in tobacco and tobacco extracts, in which I used the Kissling method and also the modification proposed by Winton and the Lloyd method. It seems that the Lloyd method gave as good results as either of the others and was much more rapid and easily worked and much better adapted to technical work. In this connection I have been trying luteol as indicator in titrating the nicotin, and I believe it is better for this purpose than litmus, being apparently much sharper in its indications.

A. L. Winton calls attention to the method for the determination of formaldehyde, which is described on page 143 of the Connecticut Report for 1899.

With these exceptions, the replies received related to the determination of arsenic in arsenicals, especially paris green, the separation of the arsenic and the allied determinations of copper, acetic acid, water-soluble arsenic, and lead.

For the separation of the arsenic from copper, Goessman and Avery precipitate the copper as suboxid; Goessman (alternate method) and Myers precipitate the mixed sulphids and separate with sodium sulphid, and Peter and Patterson determine the arsenic by precipitation as magnesium pyroarsenate without separation, the copper passing into the filtrate. C. H. Jones also precipitates directly or with previous separation with ammonium molybdate. He also communicates the separation of arsenic from lead, which is particularly applicable in the analysis of arsenate of lead.

The determination itself of the arsenic is by oxidation and precipitation as magnesium pyroarsenate in the laboratories of Peter, Goessman, Fulmer, Patterson, Hardin, and Jones; S. Avery uses the volumetric method of Thorn Smith, and Myers and Wilson use the method proposed by the writer on page 105 of the proceedings of this association for 1899.

The determination of the copper is reported by Goessman to be as copper oxid, by Myers as copper sulphid (the method proposed in 1899), and by Fulmer and Avery by titration with potassium cyanid.

Professor Goessman sends the method for the determination of the acetic acid in paris green, and the preliminary tests are from Professor Hilgard's bulletin, No. 126 of the California station.

In regard to the examination of paris green, the most extensive and systematic work that has come to the writer's notice is that of J. K. Haywood, of the U. S. Department of Agriculture, which appeared in the journal of the American Chemical Society for September, 1900, and to which the attention of the referee was called by E. E. Ewell, at that time acting chemist of the Department. It is impracticable to quote from Mr. Haywood's discussion of the several methods which he tried, for it is all of such value and importance that in order to quote at all, it would be necessary to reproduce the article in full. The methods on which he finally determines are given, and those who wish to use them are urged to consult the original article.

METHODS.

ALKALIES AND ALKALINITY.

Any of the standard methods may be used, both for the alkalies and for the alkalinity of the ash of the various soap preparations.

ARSENIC IN ARSENICALS.

Preliminary examination.

See Bulletin 126, California Agricultural Experiment Station, p. 12.

Chemical examination.

(1) *Hydrogen sulphid method.* See Bulletin 57, Division of Chemistry, United States Department of Agriculture, pp. 104, 105.

(2) *Determination as pyroarsenate.*¹ Weigh out 1 gram of material in a beaker and add 50 cc of water; digest for one-half hour in a boiling water bath with about 5 grams of caustic potash. This reduces the copper to the red suboxid. Filter, wash with water, dry the precipitate, and burn in a porcelain crucible; add a few drops of nitric acid to complete the oxidation, carefully drive off the excess of nitric acid, cool, and weigh.

Oxidize the filtrate containing the arsenic with chlorin gas, let stand for a few hours, and if chlorin gas can be detected by the smell very carefully neutralize the solution with ammonium hydrate and precipitate the arsenic with magnesia mixture; allow to stand at least two hours, filter through Gooch crucible, dry, burn, cool, and weigh, the resulting product being magnesium pyro-arsenate ($Mg_2As_2O_7$). Make the solution containing the arsenic acid with hydrochloric acid and change the arsenious acid to the arsenic acid by the addition of small quantities of chlorate of potash at frequent intervals. Drive off the excess of chlorin by repeated evaporations, make alkaline with ammonium hydrate, and precipitate with magnesia mixture.

(3) *Method of Thorn Smith, modified by J. K. Haywood.*

See Journal of the American Chemical Society, Vol. XXII, No. 9, pp. 576-578.

(4) *Estimation of copper by potassic cyanid* (Parkes and C. Mohr). See Sutton's Volumetric Analysis, pp. 162-163.

(5) *Estimation of acetic acid.* "The figure for acetic acid in paris green is usually obtained by subtracting the sum of total arsenious oxid, water, cupric oxid, and residue left after treating with dilute hydrochloric acid, from 100."²

The following method is communicated by C. A. Goessman:

Weigh out 0.6 gram of the paris green, transfer to a flask, capacity about 400 cc. Make up a 10 per cent solution of sulphuric acid, and add about 200 cc of this solution to the substance in the flask, affix to the condenser, apply heat and distill over the acetic acid. Collect 125 cc of the distillate and titrate against N/10 soda solution, using phenol-phthalein as an indicator.

The number of cc of N/10 soda solution required to neutralize the acid will give the percentage of acetic acid.

(6) *Estimation of water-soluble arsenic.* Mr. J. K. Haywood communicates the following method:

Weigh 1 gram of paris green, transfer to a 500 cc flask, add 400 cc water at room temperature, and shake with a rotary motion. Allow the flask to stand at room temperature for from twelve to fourteen days, shaking occasionally each day. Finally complete the volume to 500 cc, filter through a dry filter, take 100 cc of the filtrate, and determine the arsenious oxid.

(7) *Estimation of arsenate of lead.* Mr. C. H. Jones, chemist of the Vermont Agricultural Experiment Station, writes as follows:

Below find the outline of a method given me by Mr. F. J. Smith, late chemist of the Massachusetts Gypsy Moth Commission, which is particularly applicable in the analysis of arsenate of lead. I have used it in the estimation of arsenic in the above-named material, and it is apparently satisfactory.

Take 0.5 gram of material and mix in porcelain dish with two parts sodium carbonate and one part powdered sulphur. Fuse at a low temperature for about thirty minutes, and then increase heat if need be until sulphur fumes are driven off. Cool, digest in warm water until dissolved. The lead and arsenic are as sulphids in alkaline solution—arsenic soluble, lead insoluble. Filter, using Gooch crucible, and wash with ammonium acetate solution (3 grams to 200 of water) to prevent lead from running through. The lead in the precipitate may be estimated as desired. Filtrate is evaporated to dryness, oxidized with fuming nitric acid and a trifle of potassium chlorate and hydrochloric acid, and precipitated in ammoniacal solution with magnesia mixture directly (preferred) or with molybdic solution as with phosphoric acid.

It will be noticed that I have given in this compilation the methods in their entirety, as communicated to me or referred to the publications where they are printed. This is especially noticeable in the case of the methods for the analysis of the arsenicals. Perhaps a more systematic, and hence a more scientific, plan would

¹ As communicated to the referee by C. A. Goessman.

² Journal of the American Chemical Society, Vol. XXII., No. 9, p. 578.

have been to compile all the separations as such, and follow with the methods for the actual determinations, but I have not done so, as this would destroy the individuality of the contributions, and in many cases the particular method of determination employed follows the method of separation as a natural sequence. I submit this compilation to the Association of Official Agricultural Chemists as representing the opinion of its workers, and would respectfully recommend that they all be adopted provisionally for trial and comparison in actual analytical work in the same manner as has in the past so perfected our methods of fertilizer analysis.

POTASSIUM CYANID.

Determine purity by Liebig's volumetric method. It is based on the fact that when a solution of silver nitrate is added to an alkaline solution containing cyanogen, with constant stirring, no permanent precipitate of silver cyanid occurs until all the cyanogen has combined with the alkali and the silver to form a soluble double salt (in the presence of potash, for example, KCy , AgCy). If the slightest excess of silver over and above the quantity required to form this combination be added, a permanent precipitate of silver cyanid occurs, the double compound being destroyed.¹ If, therefore, the silver solution be of known strength, the quantity of cyanogen present is easily found, 1 eq. of silver in this case being equal to 2 eq. of cyanogen.

FORMALIN.

Qualitative: Use the tests for formaldehyde in milk described on page 113 of the proceedings of the convention of this association in 1897 (Bulletin No. 51, Division of Chemistry).

Quantitative: Method of Blank and Finkenbeiner,² of which the following is an abstract:

(1) Three grams of the solution are weighed off into a tall Erlenmeyer flask containing 25 to 30 cc of double normal sodium hydroxid; 50 cc of pure 2.5 to 3 per cent hydrogen peroxid solution are then gradually added during a space of 3 minutes, through a funnel placed in the neck of the flask to prevent spurting. After standing two or three minutes the funnel is washed with water and the unused sodium hydroxid titrated with double normal sulphuric acid, using litmus as indicator.

When analyzing solutions containing less than 30 per cent of formaldehyde the mixture must be allowed to stand 10 minutes after adding the hydrogen peroxid to complete the reaction.

The percentage of formaldehyde is found by multiplying the number of cubic centimeters of soda solution used by 2 when 3 grams of substance are taken.

Acetaldehyde reacts much more slowly with the reagent than formaldehyde, and it is doubtful whether the reaction is quantitative. Paraldehyde reacts still more slowly with hydrogen peroxid, even in the presence of a trace of ferrous salt. Benzaldehyde is acted upon more quickly, particularly in presence of ferrous sulphate, and a considerable evolution of gas takes place, but a long time is needed to complete the reaction.

(2) Method in use at the Connecticut Agricultural Experiment Station. (See Twenty-third Annual Report, 1899, p. 143.)

NICOTIN.

(1) *Kissling's method.* See Wiley, Principles and Practice of Agricultural Analysis, Vol. III, p. 606.

¹ "So fast is this double combination that when sodic chlorid is present, no permanent precipitate of silver chlorid occurs until the quantity of silver necessary to form the compound is slightly overstepped." Sutton's Volumetric Analysis, 1890, p. 173, which see for further details; also Fresenius Quantitative Analysis, 1886, p. 450.

² Ber. 31, 2979.

(2) *Winton's modification*. See Bulletin 56, Division of Chemistry, United States Department of Agriculture, p. 125.

(3) *Lloyd's method*. See Bulletin 56, Division of Chemistry, United States Department of Agriculture, p. 114.

The methods for fungicides and insecticides presented by Mr. Voorhees in his report were adopted provisionally for trial and comparison in actual analytical work.

The report of the committee on abstracts was now called for, and, in the absence of Mr. Allen, was read by Mr. Beal, as follows:

REPORT OF THE COMMITTEE ON ABSTRACTING.

MR. PRESIDENT AND MEMBERS OF THE ASSOCIATION:

The work of the committee on abstracting literature relative to methods and apparatus for agricultural analysis has progressed during the past year with a fair degree of regularity. As soon as possible after the designation of the committee, the journals accessible to its members were ascertained and the following allotment was made by the chairman:

E. W. Allen.... Jour. Landw.; Milch Ztg.; Ztschr. Untersuch. Nahr. u. Genussmtl.; Ztschr. Physiol. Chem.; Chem. Centbl.; abstracts in Analyst and Chem. Ztg. Report.

J. T. Anderson... Chem. Ztg.; Jour. Chem. Soc. [London].

W. H. Beal..... Compt. Rend.; Bul. Soc. Chim. Paris; Oesterr. Chem. Ztg.; Ztschr. Angew. Chem.; Ber. Deut. Chem. Gesell.; abstracts in Jour. Chem. Soc. [London].

R. E. Blouin.... Bul. Assoc. Chim. Sucr. et Distill.; Jour. Amer. Chem. Soc.

W. F. Hand.... Chem. News; Jour. Soc. Chem. Ind.

G. W. Shaw.... Amer. Chem. Jour.; Analyst.

Harry Snyder... Bul. Assoc. Belge Chim.; Rev. Chim. Analyt. et Appl.

J. P. Street.... Landw. Vers. Stat.; Jour. Franklin Inst.

C. B. Williams... Ztschr. Analyt. Chem.; British Food Jour.

It is inevitable that at times the work assigned to individual members of the committee will accumulate, since most of the members are actively engaged in laboratory work and are likely to have unusual pressure of work upon them at certain periods in the year. If the work accumulates to any considerable extent, it is likely to become a burden upon the abstractor and to result in the oversight of part of the articles. To avoid this and to keep the work up to date, the plan has been followed, as in the past, of having such numbers of journals as have become two months in arrears abstracted in the Office of Experiment Stations. The object of this, as will be evident, is merely to prevent the work becoming burdensome and to avoid gaps.

During the past year there has been quite the usual amount of investigation on chemical methods, some of which it may not be without interest to mention very briefly, with the reference to the place of publication of the abstract in Experiment Station Record (E. S. R.)

The discovery of the occurrence of perchlorate in Chile saltpeter and its injurious effect upon crops has brought out a considerable number of articles on the frequency and extent of its occurrence, its injurious effects, and the methods for estimating it (E. S. R., XI, p. 110).

The constitution of the ammonium-magnesium phosphate of analysis has been reported upon in a valuable contribution by F. A. Gooch and Martha Austin (E. S. R., XI, p. 107).

F. P. Veitch has investigated the direct determination of available phosphoric acid and pointed out the cause of the discrepancy between the results by the Ross direct method and the official method (E. S. R., XII, p. 306).

E. Hotter has studied the solubility of Thomas slag in citric-acid solutions and recommends the use of a solution not stronger than 4 per cent (E. S. R., XI, p. 109).

Aschman has described a method for determining the total phosphoric acid in Thomas slag, using nitro-sulphuric acid as a solvent (E. S. R., XI, p. 507).

W. Hoffmeister has reported the results of studies with various slags, which indicate that his humic-acid method furnishes a reliable means of determining the relative value of different phosphates (E. S. R., XI, p. 1104).

Maxwell has reported his investigations on the estimation of available lime, potash, and phosphoric acid in soils by means of aspartic acid (E. S. R., XI, p. 507).

J. Plot has described the determination of phosphoric acid available as plant food in soils and fertilizers by means of a solvent, which it is claimed closely resembles beet juice in respect to salts. He gives a formula for its preparation (E. S. R., XII, p. 211).

Hess and Campbell have reported a new method for the direct determination of alumina in the presence of iron, manganese, calcium, and magnesium (E. S. R., XI, p. 611).

H. Lasne has given detailed directions for microscopical and chemical examination of bone superphosphate for purposes of detecting adulteration (E. S. R., XI, p. 104).

C. M. Van Deventer has proposed a new method of determining nitrates based upon the formation of brown solutions by the reaction between nitrates and ferrous sulphate (E. S. R., XI, p. 811).

E. W. Bell described a method for estimation of potash in fertilizers, soils, and vegetable substances, which is reported as having given very satisfactory results (E. S. R., XI, p. 109).

The determination of humus in soils has been described by C. Aschman and H. Faber (E. S. R., XI, p. 110).

A new method for the determination of clay in soils, by F. Poquillon, is described as fully as accurate and much more rapid than former methods (E. S. R., XI, p. 903).

Thorn Smith has proposed a method for the estimation of arsenic in paris green which reduces the time required for analysis to less than an hour (E. S. R., XI, p. 614).

T. B. Stillman has studied the variation in the composition of paris green and proposed a scheme for its analysis (E. S. R., XI, p. 22).

O. Kellner, F. Herring, and O. Zahn have tested König's method for the determination of pentosan-free crude fiber (treatment in an autoclave with sulphuric acid and glycerin). The crude fiber by this method contained from 2 to 3 per cent of pentosans, whereas that by the Weende method contained from 16 to nearly 20 per cent (E. S. R., XI, p. 704).

A critical study of the inverting power of tartaric, citric, and oxalic acids upon sucrose has been reported by H. Gillott (E. S. R., XI, p. 20).

The determination of cane sugar in condensed milk has been investigated at considerable length by L. Grünhut and S. H. Riiber (E. S. R., XII, p. 211).

P. Chapelle has described a new method for the gravimetric determination of reducing sugars, based upon the use of the centrifuge (E. S. R., XII, p. 106).

M. D. Crispo has described a new process for the determination of starch, based upon the polarization of a solution of the starch in potash, which, however, has not been tested on cereals (E. S. R., XI, p. 417).

Directions for the qualitative and quantitative examination of flour, with especial reference to adulteration with corn meal, have been given by H. Kraemer (E. S. R., XI, p. 211).

J. K. Haywood has worked out a modification of the Brücke method for determining glycogen, which is sufficiently accurate for distinguishing horse meats from other meats (E. S. R., XII, p. 107).

Upon the subject of milk and food preservatives, especially boric acid and formaldehyde, there have been a large number of articles treating of the extent of their use, the means of detection, and their effect upon the human system (E. S. R., XI, pp. 184, 212, 213, 312, 418, 419, 482, 575, 587, 618, 705, 769, 904, 960, 962, 984; XII, pp. 213, 214, 280).

There has been a large amount of work on the detection of adulteration in butter, the range in the constants with the change of period of lactation and with the change of feed (E. S. R., XI, pp. 111, 975; XII, pp. 18, 181, 308).

Among the new apparatus, perhaps the most important is that for ash determination described by Shuttleworth (E. S. R., XI, p. 304) and modified by Tucker (E. S. R., XI, p. 506). This apparatus, with the use of acetate of lime to prevent the formation of silicates insoluble in hydrochloric acid, places in the hands of the analysts a means for increasing the accuracy of ash determination and ash analysis. The investigations which these two gentlemen have reported show how relatively large may be the losses and the errors by the more common and cruder methods of incinerating materials.

An electrical method for the determination of nitrogen in organic substances has been described by Budde and Schou (E. S. R., XI, p. 306), in which the digestion in the Kjeldahl flask is accelerated by means of an electric current. The method does away with the addition of any foreign substance to promote the reduction. The results reported by these authors show a rather closer agreement with the theoretical than the ordinary Kjeldahl method gave.

A. W. Blyth has devised an apparatus in which nitrites and nitrates, either singly or together, can be determined as nitric oxid by means of ferric chlorid (E. S. R., XI, p. 21).

J. L. Hills, B. O. White, and C. H. Jones have reported the results of investigations on the experimental error involved in sampling crops for analysis (E. S. R., XI, p. 307).

C. A. Browne, jr. (E. S. R., XI, pp. 308, 615, 616) has given a valuable contribution to the chemistry of butter fats, dealing with the physical and chemical constants, the chemical composition, and the chemistry of rancidity in butter fat.

T. B. Osborne has continued his valuable investigations on the proteids of agricultural plants and products (E. S. R., XI, p. 309; XII, No. 6).

H. Droop Richmond has given us a very valuable contribution to the chemistry of dairy products in his *Dairy Chemistry: A Practical Handbook for Dairy Chemists and Others Having Control of Dairies* (E. S. R., XI, p. 618).

These are only a few of the papers relating to methods of agricultural analysis which have been noted by the abstract committee and abstracts published currently in the Experiment Station Record.

E. W. ALLEN,
J. T. ANDERSON,
W. H. BEAL,
R. E. BLOUIN,
W. F. HAND,
G. W. SHAW,
J. P. STREET,
H. SNYDER,
C. B. WILLIAMS.

Committee.

The report of the committee on fertilizer legislation was asked for at this stage of the proceedings, but no report was made, the committee having taken no new action in the matter.

Mr. Huston suggested that the report already submitted was a good starting point.

Mr. Wiley agreed to have reprints made of the report.

The PRESIDENT. The Association of Commissioners of Agriculture of the South presented the views of this committee and urged the commissioners to adopt them. The chemists should seek the aid of these gentlemen.

The report of the committee on recommendations was then presented.

REPORT OF COMMITTEE ON RECOMMENDATIONS.

SUGAR.

The committee recommends that the referee for the ensuing year study the method of Clerget and the German method.

Adopted.

REPORT OF COMMITTEE ON VOLUMETRIC STANDARDS.

The report of the committee on volumetric standards was presented by Mr. Ewell, at the request of the president. It consisted in a statement of the progress made in the movement to establish a national standardizing bureau, and proposed the following resolutions, which were adopted:

Resolved, That the Association of Official Agricultural Chemists most heartily indorses the movement in progress for the establishment in this country of a national standardizing bureau, and hereby declares that the absence of facilities such as would be provided by the proposed bureau has seriously hampered the work of this association, owing to the difficulty of obtaining in this country, with official certificates of accuracy, the flasks, burettes, pipettes, weights, thermometers, polariscopes, and other apparatus needed in the work of official chemists. The use of apparatus which bears the official stamp of the Government would eliminate one element of dispute in commercial analyses, thus preventing the expense of litigation, and would in general increase the value of the work of this association by facilitating the attainment of uniform results.

Be it further resolved, That copies of these resolutions be forwarded by the members of this association to the Senators and Representatives representing their respective States in Congress.

B. W. KILGORE, *President*.

H. W. WILEY, *Secretary*.

Mr. Williams here presented a method of marking flasks by the use of paraffin and beeswax.

Mr. Frear then presented the report of the committee on food standards, as follows:

REPORT OF COMMITTEE ON FOOD STANDARDS.

On behalf of your committee on pure-food standards I would report that a meeting was held in Washington in March last, at which were present most of the members of the general committee, as well as a large number of the referees, who are collaborating with them and whose counsel in the final deliberations of the committee was most helpful.

The general plan of operation adopted by your committee makes it necessary to delay the announcement of the details of the work until the latter shall have reached some measure of completeness. This plan involves the preparation by the several referees and the consideration by your committee of correct definitions and classifications of food materials and the consideration of proposed standards in the light of American experience, as shown by the work of your committee and the referees and by compilations of existing data from materials of known character.

This preliminary work done, it is planned that the definitions and various suggested standards for the several classes of products be submitted to competent representatives of the respective trade interests concerned for criticism and suggestion before any publication of the conclusions is made by the committee, to the end that no overhasty action to the unfair detriment of any interest be taken and that the completed work may find general acceptance on the ground of its scope, accuracy, fairness, and unity.

To give some general notion of the work done, I may, however, state that definitions and standards have been tentatively adopted for the following classes of products:

Definitions: Meats (fresh, preserved, pickled, salted, and smoked); sausages; lard; milk; skim milk; cream; butter; buttermilk; cheese; whey; koumys; condensed milk; pasteurized milk; ice cream; fruit butter; jam, marmalade and fruit jelly; sugar; maple sugar; molasses; sirup; glucose sugar; glucose sirup.

Standards: Lard; milk; butter fat; butter; cream; cheese; skim milk; condensed milk; fruit butter; jam, marmalade preserves, and fruit jelly; sugar; molasses; sirup; glucose sugar; glucose sirup; flavoring extracts of cinnamon, peppermint, lemon, and orange.

Sets of definitions and standards have been offered for consideration by the referees, but not yet formally acted upon by the committee, for the followings substances:

Definitions: Salt; olive oil; spices; tea; coffee; cocoa; chocolate; fruit juices; fermented cider; perry; wines; vinegars; malted liquors; spirituous liquors.

Standards: Salt; olive oil; spices; tea; coffee; cocoa; chocolate; fermented cider; dry white wine; dry red wine; fortified sweet wine; cider vinegar; wine vinegar; malt vinegar; malt beer; malt extracts; whisky; brandy; rum; gin.

In connection with this work the following collections of data, in many instances involving original research, have been made for the specific work of this committee, and those not yet presented for publication are now nearly ready for the press:

- (1) *Lard*.—Results of analyses of lard used for cheese filler. By J. B. Weems.
- (2) *Milk products*.—Milk: Compilation of averages of normal milk varying between 3 and 5 per cent of butter fat, representing 5,552 samples. By L. L. Van Slyke.
- (3) *Cocoonut oils*.—Results of analyses of cocoonut oils used as butter surrogate. By J. B. Weems.
- (4) *Grains and their products*.—Computation of maximum, minimum, and averages representing the composition of barley meal and pearl barley; buckwheat products—flour, self-rising buckwheat, prepared buckwheat, buckwheat farina; corn products—meal, granular, unbolted, flour, velvet meal, cerealine, hominy, New Mexico corn, pop corn (raw and popped); prepared infant and invalid foods (16 kinds); oat breakfast foods (15 kinds); macaroni and vermicelli; wheat breakfast foods (26 kinds); wheat flour (20 kinds); bread and other baked flour products (51 kinds); starches (2 kinds). By W. O. Atwater.

(5) *Cattle foods*.—Compilation of 636 complete or partial analyses of 16 kinds of concentrated cattle foods. By J. B. Lindsey.

(6) *Fruits and vegetables*.—A compilation of analyses of market fruit butters, jellies, preserves, and marmalades, representing 38 samples. By H. A. Weber.

(7) *Condiments*.—A compilation showing (1) standards recommended for various species by Vogl and Hanausek, published in Codex Alimentarius Austriacus; (2) results of 206 American analyses of species and their adulterants, the majority of them by the compiler and his associates, together with description of microscopic and chemical methods of investigations. By A. L. Winton.

(8) *Fruit extracts*.—Results of analyses of fruit extracts for table use. By H. A. Weber.

(9) *Salt*.—Compilation of 87 American analyses of table, dairy, and packing salts, largely made by the compiler for use of the committee. By F. W. Woll.

(10) *Olive oil*.—Compilation of the properties of olive oil, European and American, and of its salad-oil substitutes and adulterates, including the index of refraction, specific gravity, thermal degree, viscosity of soap solution, iodine number, saponification value, melting point of fatty acids, behavior under special tests: Brullé, Bechi, Schneider, Renard, Baudouin, and Hauchecorne, including detailed results. By G. E. Colby.

(11) *Cotton-seed oil*.—Results of analyses of cotton-seed oil. By J. B. Weems.

(12) *Beverages and vinegar*.—Compilation of American and European results of analyses of apple juice, fermented cider, cider and spirit vinegar. By William Frear.

Your committee desires to express its gratitude for the able and painstaking collaboration of the several referees and their associates in this work, to whom the progress it has thus far made is chiefly due.

The committee also desires to express to Hon. James Wilson, Secretary of Agriculture, its appreciation of his cordial indorsement of their work and the aid he has given in facilitating correspondence by appointing the members of the committee and the referees in charge of main classes of subjects special agents of the United States Department of Agriculture.

In closing this report of its work your committee requests that it be continued for further effort toward the end proposed.

Respectfully submitted in behalf of the committee.

WILLIAM FREAR,
Chairman.

The report was adopted and the committee continued for the ensuing year.

The following resolution was presented by Mr. Frear for the committee on pure-food standards:

Resolved, That the Association of Official Agricultural Chemists of the United States hereby reaffirms its conviction of the urgent necessity for national legislation on broad lines for the prevention of adulteration of foods and drugs that are subjects of interstate commerce and commerce with foreign lands, for the better protecting of the health of our citizens and the legitimate interests of producers and dealers, and for the national reputation of business honor, and most earnestly and respectfully urges upon the Congress of the United States the present enactment of the measure now on the Calendar of the House of Representatives with a favorable recommendation, after long and careful consideration of the Committee on Interstate and Foreign Commerce, which is essentially the bill introduced in the present Congress in the House by Hon. Marriott Brosius, of Pennsylvania, known as H. R. 9677, and in the Senate by Hon. H. A. Hansbrough, numbered 2222 and now under consideration by the appropriate committee of that body.

Resolved further, That the secretary of the association be directed to convey to the proper officers of the Congress of the United States copies of these resolutions.

Adopted.

The report of the committee on resolutions was then called for, which was presented, as follows:

REPORT OF COMMITTEE ON RESOLUTIONS.

Resolved, That the Association of Official Agricultural Chemists return cordial thanks to the Secretary of Agriculture, the authorities of Columbian University, and the governing board of the Cosmos Club for the courtesies they have shown to the association.

Adopted.

The PRESIDENT. I am sorry we have not the blackboard before us, so we can see the roll of this meeting. It numbers upward of 85. This is the largest attendance this association has ever had. Dr. McMurtrie, president of the American Chemical Society, in speaking to me to-day, said, "If we can have 100 members present at the Chicago meeting of the American Chemical Society, I shall consider that we are very fortunate." Inasmuch as we are but a small part of the broad field of chemistry, I feel that the good showing at this meeting is a matter for congratulation. Another matter has come before us at this time. We have had our friends from across the border come to the association with their troubles. They recognize the value of our methods. We are glad to hear from the report of our secretary that our methods are appreciated and recognized, not only abroad, but also at home their great value is seen. These things make me appreciate all the more the great honor you have done me in selecting me to preside over the seventeenth annual convention, and I thank you most heartily and sincerely for this compliment.

If there is no further business, I shall declare the seventeenth annual meeting of the Association of Official Agricultural Chemists adjourned sine die.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1901.

PRESIDENT.

Mr. L. L. VAN SLYKE, Geneva, N. Y.

VICE-PRESIDENT.

Mr. H. J. WHEELER, Kingston, R. I.

SECRETARY.

Mr. H. W. WILEY, Washington, D. C.

ADDITIONAL MEMBERS OF EXECUTIVE COMMITTEE.

Mr. W. R. PERKINS, Agricultural College, Miss.

Mr. F. W. TRAPHAGEN, Bozeman, Mont.

REFEREES.

Phosphoric acid.—Mr. H. K. Miller, Lake City, Fla.

Nitrogen.—Mr. W. R. Perkins, Agricultural College, Miss.

Potash.—Mr. C. L. Hare, Auburn, Ala.

Soils.—Mr. M. E. Jaffa, Berkeley, Cal.

Dairy products.—Mr. J. A. LeClerc, Geneva, N. Y.

Foods and feeding stuffs.—Mr. W. H. Krug, Washington, D. C.

Liquor and food adulteration.—Mr. W. D. Bigelow, Washington, D. C.

Sugar.—Mr. E. E. Ewell, Washington, D. C.

Tannin.—Mr. W. K. Alsop, 26 Ferry street, New York.

Insecticides.—Mr. Louis A. Voorhees, New Brunswick, N. J.

Ash.—Mr. G. S. Fraps, Raleigh, N. C.

ASSOCIATE REFEREES.

Phosphoric acid.—Mr. C. H. Jones, Burlington, Vt.

Nitrogen.—Mr. Fred W. Morse, Durham, N. H.

Potash.—Mr. H. B. McDonnell, College Park, Md.

Soils.—Mr. F. P. Veitch, Washington, D. C.

Dairy products.—Mr. George Cavanaugh, Ithaca, N. Y.

Foods and feeding stuffs.—Mr. C. A. Browne, jr., State College, Pa.

Liquor and food adulteration:

(1) Meat and fish.—W. D. Bigelow, Washington, D. C.

(2) Fats and oils, not including dairy products or their substitutes.—L. M. Tolman, Washington, D. C.

(3) Cereal products.—A. McGill, Ottawa, Canada.

(4) Infants' and invalids' foods.

(5) Saccharine products, including confectionery.—A. E. Leach, Boston, Mass.

(6) Vegetables—canned, dried, or otherwise preserved.—L. S. Munson, Washington, D. C.

- (7) Tea, coffee, and cocoa.
- (8) Spices and condiments.—A. L. Winton, New Haven, Conn.
- (9) Vinegar.—William Frear, State College, Pa.
- (10) Flavoring extracts.—A. S. Mitchell, Milwaukee, Wis.
- (11) Fruit products.—L. M. Tolman and L. S. Munson, Washington, D. C.
- (12) Fermented and distilled liquors.
- (13) Baking powders and baking powder chemicals.—A. L. Winton, New Haven, Conn.
- (14) Preservatives.—W. M. Allen, Raleigh, N. C.
- (15) Dyes.—L. M. Tolman, Washington, D. C.
- Sugar*.—Mr. G. L. Spencer, Washington, D. C.
- Tannin*.—Mr. W. H. Teas, Ridgway, Pa.
- Insecticides*.—Mr. J. K. Haywood, Washington, D. C.
- Ash*.—Mr. E. W. Magruder, Richmond, Va.

COMMITTEE ON ABSTRACTING.

Mr. E. W. Allen, Washington, D. C., Chairman.
 Mr. W. H. Beal, Washington, D. C.
 Mr. J. T. Anderson, Auburn, Ala.
 Mr. J. P. Street, New Brunswick, N. J.
 Mr. W. F. Hand, Agricultural College, Miss.
 Mr. Harry Snyder, St. Anthony, Minn.
 Mr. C. B. Williams, Raleigh, N. C.

COMMITTEE ON FOOD STANDARDS.

Mr. Wm. Frear, State College, Pa., Chairman.
 Mr. H. W. Wiley, Washington, D. C.
 Mr. H. A. Weber, Columbus, Ohio.
 Mr. M. A. Scovell, Lexington, Ky.
 Mr. E. H. Jenkins, New Haven, Conn.

COMMITTEE ON FERTILIZER LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., Chairman.
 Mr. B. W. Kilgore, Raleigh, N. C.
 Mr. H. B. McDonnell, College Park, Md.
 Mr. H. A. Huston, Lafayette, Ind.
 Mr. B. B. Ross, Auburn, Ala.

COMMITTEE ON VOLUMETRIC STANDARDS.

Mr. B. W. Kilgore, Raleigh, N. C., Chairman.
 Mr. C. L. Penny, Newark, Del.
 Mr. E. E. Ewell, Washington, D. C.
 Mr. G. C. Caldwell, Ithaca, N. Y.
 Mr. H. W. Wiley, Washington, D. C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members, and to have all privileges of membership, save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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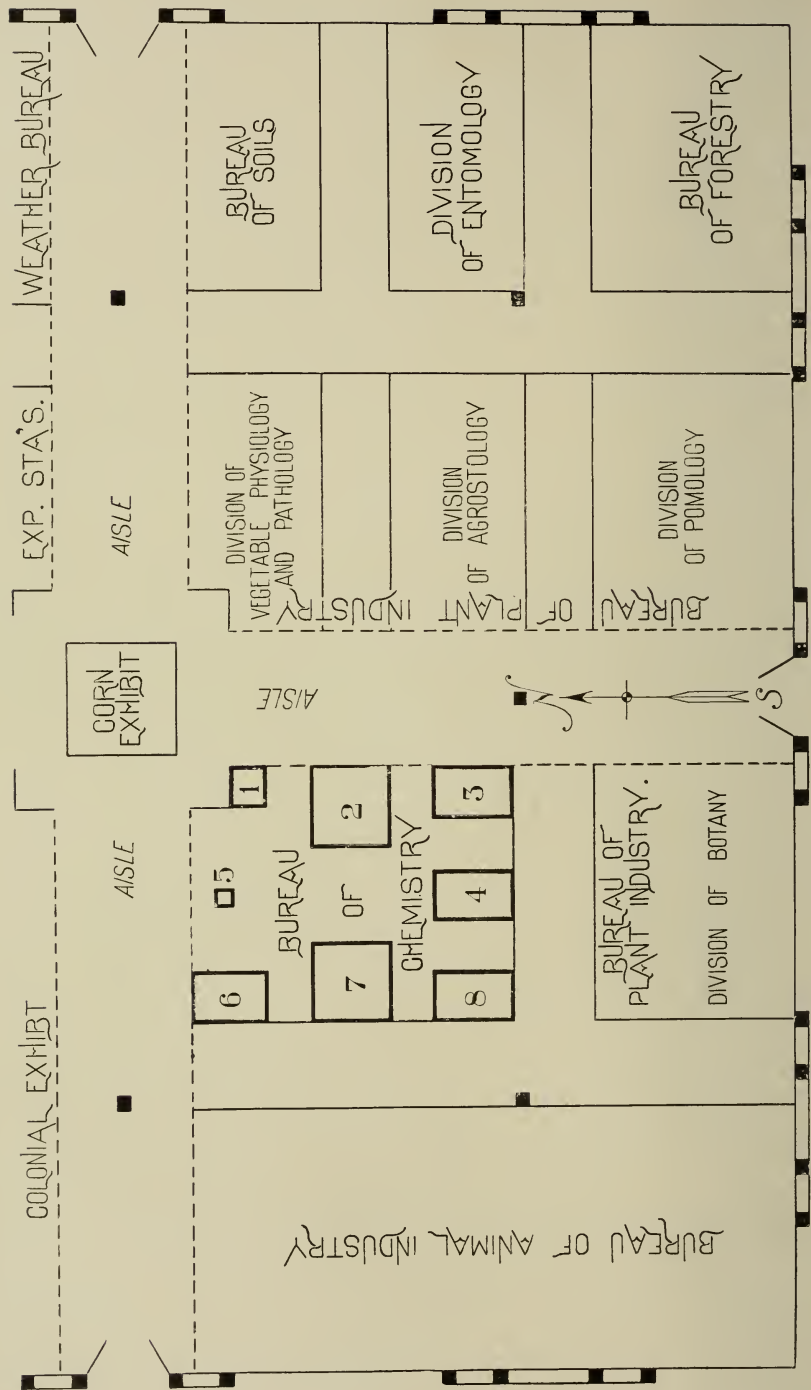


DIAGRAM OF THE FLOOR PLAN OF THE NORTH WING OF THE UNITED STATES GOVERNMENT BUILDING, PAN-AMERICAN EXPOSITION.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY.

EXHIBIT

OF THE

BUREAU OF CHEMISTRY

AT THE

PAN-AMERICAN EXPOSITION,

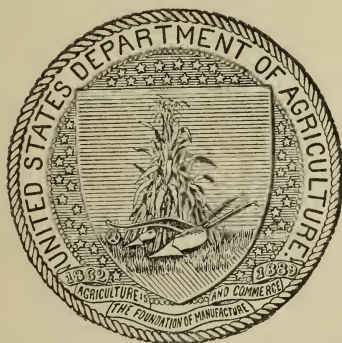
BUFFALO, NEW YORK, 1901.

PREPARED UNDER THE DIRECTION OF

HARVEY W. WILEY,
CHIEF OF BUREAU,

BY

E. E. EWELL, W. D. BIGELOW, AND LOGAN WALLER PAGE.



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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., July 1, 1901.

SIR: I have the honor to transmit to you herewith the manuscript of a description of the exhibit of the Bureau of Chemistry at the Pan-American Exposition, Buffalo, N. Y., 1901, with the request that it be published as Bulletin No. 63 of the Bureau of Chemistry.

H. W. WILEY, *Chemist.*

Hon. JAMES WILSON,
Secretary of Agriculture.

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EXHIBIT OF THE BUREAU OF CHEMISTRY AT THE PAN-AMERICAN EXPOSITION, BUFFALO, NEW YORK, 1901.

INTRODUCTION.

The exhibit of the Bureau of Chemistry will be found in the central part of the north wing of the Government building. It has been so planned as to illustrate three of the important features of the work of the Bureau, namely, the study of pure and adulterated foods, the beet-sugar industry, and the testing of road-making materials. It was impracticable to include in the exhibit a number of lines of investigation which are now in process or which have been completed during recent years. The results of many of these have been published, and are shown in a collection of publications which forms a prominent feature of the exhibit. These will be found on the table marked "1." (See Pl. I, frontispiece.) Since 1883, 62 bulletins and 7 small pamphlets, designated as circulars, have been issued, containing in all 7,989 pages. Numerous contributions have been made by members of the Bureau to the Yearbook of the Department of Agriculture. The reports of sugar-beet investigations since 1897 have been published as a part of the special report to Congress on this subject. The results of the food investigations hitherto published are contained in the nine parts of Bulletin 13 (1,374 pages). In addition to the work reported in the regular publications of the Bureau, a considerable amount of research work has been done which has been reported from time to time in various scientific periodicals.

Much of the time of the working force of the Bureau is required for cooperative work carried on with or at the request of other branches of the Department of Agriculture and the other Executive Departments of the Government.

EXHIBIT OF PURE AND ADULTERATED FOODS.

By W. D. BIGELOW.

In this exhibit it is desired to illustrate principles and not to call attention to individual frauds. Interest would have been added had it seemed best to display all the samples under their original labels. It is apparent, however, that such a policy would have been unjust to all manufacturers of pure foods whose goods were not exhibited,

and would have discriminated against adulterated foods exhibited in favor of those omitted. It seemed essential, therefore, to transfer all samples to uniform glass bottles, and thus avoid discrimination with its consequent injustice. On approaching the exhibit its most conspicuous feature is the series of silk fabrics, dyed with the aniline coloring matter contained in a large assortment of foods and food adjuncts. The method by which these fabrics were dyed precludes the possibility of the presence of any vegetable coloring matter. The color of the fabric is due in every case to aniline color added to the food as an adulterant.

CASE 2.¹

The top shelf contains samples of substances which are used to adulterate alcoholic liquors and vinegar. The three samples of vinegar flavor, though given different names, are identical in composition. It is intended that this article shall be added to dilute acetic acid, which is then artificially colored and the product sold as cider vinegar or wine vinegar. Although the mixture thus formed does not closely resemble pure vinegar, it is sold in large quantities in localities where food laws are not enforced.

It is well known that alcoholic liquors soften with age, and various devices are employed to give new liquors the flavor of old. Among other methods may be mentioned the addition of various chemicals, of which the aging oil on this shelf is an illustration.

The alcoholic liquors on the market are sometimes artificially prepared by flavoring and coloring diluted alcohol. The peach-brandy essence shown on the same shelf is sold for the manufacture of peach brandy. It is directed to mix together 40 gallons of proof spirit, one-half pound of this essence, 1 quart of sugar sirup, and a sufficient amount of coloring matter advertised by this same firm to give the desired color.

The bead oil which is exhibited on the same shelf is a solution of soap which is sometimes added to distilled liquors to produce a "bead," and thus give the article the appearance of age.

Hop extract, a sample of which is shown, is often obtained by extracting inferior hops, and the exhausted residue is then sometimes placed on the market as untreated hops.

Flavoring extracts and soda-water sirups.—The second and third shelves of this case are devoted to an exhibit of flavoring extracts, soda-water sirups, etc. The labels are, for the most part, a sufficient explanation of the samples on these shelves, but especial attention may be called to the practice of substituting tonka beans for vanilla beans in the preparation of this extract. The use of artificial flavoring material or mixtures of cheaper substances in place of vanilla is also commonly practiced. Certainly the most reprehensible practice in

¹ See Pl. I, frontispiece.

connection with the manufacture of flavoring extracts is the use of methyl alcohol, or wood spirits, as a solvent in place of ordinary alcohol. Cases of death resulting from the use of lemon extract containing wood spirits are of common occurrence, and although such results follow the use of extracts as a beverage instead of the use for which they are intended, it is still true that an article of such toxic properties should never be added to food or food adjuncts. In this connection it should be stated that the poisonous properties of wood spirits are not due solely to the methyl alcohol they contain, but to impurities that always occur in the commercial article.

Sweetening materials and saccharine foods.—On the fourth shelf of this case are exhibited samples illustrating the most prevalent forms of adulteration used in connection with sugar and sweetening materials. One sample each of granulated sugar and “A” sugar is shown. These substances are rarely adulterated. The prevalent notions of admixture of sand and clay are entirely without foundation.

Adulterations do occur in some cases, however, and the sugars adulterated with glucose and saccharin exhibited on this shelf are now sold to a limited extent in various parts of the country.

Saccharin is placed on the market in several degrees of purity, the highest of which is about five hundred times as sweet as sugar. It is an artificial preparation manufactured from coal tar and possessing an intensely sweet taste. Saccharin passes through the body unchanged. It is therefore of value for sweetening foods for patients who are not allowed to receive sugar, but it has no food value whatever.

Five samples of glucose and grape sugar, illustrating the principal grades on the market, are shown on the same shelf. The liquid or sirupy preparations are commercially known as glucose, while those that are prepared in the solid state, though obtained from the same source, are known as grape sugar or glucose sugar. This product is used extensively with all varieties of sweetened foods.

A large percentage of the various table sirups on the market and many strained honeys are sweetened partly or entirely with glucose. Several samples of these articles containing glucose are exhibited on the same shelf.

Glucose is used in the manufacture of jellies, jams, and marmalades. The claim is commonly made that its use in such products is due not so much to its cheapness as to the fact that preparations containing it have less tendency to crystallize or “candy” than those prepared entirely with cane sugar.

Cacao and cacao preparations.—On the fifth shelf of this case two cacao or cocoa pods are exhibited, from one of which a segment has been removed, showing the cacao seed or cocoa bean in place. Five samples of these seeds grown in different localities are also exhibited.

Chocolate is prepared by grinding the shelled cacao seed or cocoa bean, mixing the ground product to a pulp, and molding. It may be

either sweetened or unsweetened. The same product is used in the preparation of cacao, or cocoa, as it is commonly called, after the extraction of a portion of the fat. Both cacao and chocolate are largely adulterated, cacao shells, wheat flour, sago flour, and other materials of like nature being used for this purpose. Two samples of adulterated cacao are shown on this shelf.

Coffee.—A large part of the space on shelves 5 and 6 is occupied by samples of coffee and coffee substitutes. A number of samples of standard varieties of coffee are shown, both in the green and roasted condition. A striking similarity will be noticed in many cases between high-priced and low-priced samples of green coffee, while with roasted coffees this similarity is so great that even experts are frequently unable to distinguish them by their physical appearance. As illustrations of this may be noted the Java Peaberry, which sold at wholesale at the time this exhibit was collected at 20 cents a pound, and the Mexican and Santos Peaberry, which were sold at 13½ and 12 cents, respectively.

Again, the Bourbon Santos, which is grown in the province of Santos, Brazil, from the seed of the Arabian Mocha, closely resembles, as might be expected, the Arabian Mocha coffee. Its flavor is very different, however, and it commands a lower price in the market. These illustrations are sufficient to illustrate the principle involved, though they might be multiplied at will.

A large part of the alleged high-grade coffee sold by the trade belongs certainly to a much cheaper class of coffee than that for which it is sold. At the same time good coffee can always be obtained from reliable grocers.

On shelf 6 of this case a large number of roasted coffees are shown which illustrate even more strongly than green coffees the similarity of the high and low grades. Several mixtures of cereals, pea hulls, etc., which are sold as coffee, are also exhibited on this shelf, as well as one sample of artificial coffee beans which is composed entirely of flour.

Flour.—Owing to the firm attitude taken by American millers, the adulteration of staple brands of flour is practically unknown; still the samples of flour exhibited on shelf 6 are of considerable interest. The milling industry was seriously threatened several years ago by the extensive adulteration of wheat flour with a finely ground indian corn preparation which was sold as "flourine." The influence of this fraud in the price of flour was so disastrous that an organized fight was inaugurated by the millers and resulted in the passage of a revenue act which taxed and required the proper branding of mixtures of this nature. The result of this law was most wholesome, and the practice of adulterating wheat flour with indian corn products was quickly and effectually checked.

The ground soapstone, of which a sample is exhibited on this shelf,

was quite extensively advertised as a flour adulterant, but appears to have found but little or no sale for that purpose. It is interesting to note that the originator of this swindle is now serving a prison sentence for fraudulent use of the United States mails. One form of flour adulteration which is still extensively practised is the substitution of ordinary flour for gluten flour. It is unfortunate, but this substitution is commonly practiced. If gluten flour is of value in the diet of invalids, it is highly important that a patient should be able to obtain the article prescribed by his physician.

CASE 3.¹

Spices.—Spices probably afford a more fruitful field for adulteration than any other class of foods or food adjuncts. Some of the leading spice grinders make a practice of furnishing spices at almost any price that is desired, and the amount of foreign matter, which ordinarily consists of such materials as ground cereals, cocoanut shells, olive stones, sandal wood, mustard hulls, clove stems, linseed meal, and similar substances, is regulated according to the price of the goods sold. At the same time there are many grinders who practice no form of adulteration, and who do not handle any substance whatever, for the sophistication of their wares. It is unfortunately true, however, that a large percentage of the ground spices on the market is adulterated. Mixtures are even sold which are prepared for the express purpose of adulterating spices. Their color and physical appearance are practically identical with the spices they are intended to replace, but they contain no spicy flavor whatever. A full set of these fillers (marked "P. D.") is exhibited on the first and second shelves of this case.

The adulteration of spices, however, is not confined to the ground article. Unground pepper often receives an addition of stems, sticks, and pepper shells, which are removed in a preparation of white pepper. Cloves are often mixed with broken clove stems and pimento. Several varieties of spices are sometimes distilled with steam for the preparation of volatile oil, and the exhausted residue sold as pure spices. Examples of these various forms of adulteration are shown on the first three shelves of this case.

Even in pure, unground spices there is a great difference in grade and consequently in price. It is manifestly impossible to form a correct estimate of the wares of two grocers by comparing the prices for which they sell.

Food preservatives.—Among the most objectionable forms of food adulteration may be mentioned the use of chemical food preservatives. The compounds usually used for this purpose are salicylic, benzoic, and boric acids, and their sodium compounds, formaldehyde

¹See Pl. I, frontispiece.

and sulphites. Several others, such as ammonium fluorid, pyroligneous acid, beta naphthol, and abrastol, are used to a limited extent. These substances may be divided into two classes, those which are undoubtedly injurious, such as formaldehyde, salicylic acid, and sulphites, and those whose toxic action is disputed, like borax and benzoic acid. The addition to foods of substances belonging to the first class should be prohibited. The others should be used only with food that is so marked as to inform the purchaser of their presence. Alleged new discoveries, which are claimed to be entirely wholesome, are now extensively sold for the preservation of food. Without exception, these products consist of chemicals (often mixtures of two or more) which are well known to the scientific world, and many of which are familiar to the general public. A number of commercial preservatives, and of the chemical substances of which they are composed, are exhibited on the fourth shelf of this case.

It is claimed by those interested in their use that the amount of preservatives added to foods is so small as to be unimportant. It is certainly true, however, that the amount added sometimes greatly exceeds that which is believed to be necessary by those who favor the use of chemical preservatives. On shelf 3 of the fourth case are exhibited a series of samples of preservative chemicals which were actually recovered from foods.

Canned vegetables.—Among the most important abuses which are practiced in the preparation of canned vegetables is the use of an excessive amount of water, and of mature vegetables which are soaked before canning and placed on the market as green vegetables. Many States require that cans containing soaked goods shall have the word “soaked” printed in conspicuous type on the label. The tendency of some firms to evade pure-food legislation was illustrated on the original labels of some of the samples exhibited on the shelves 4 and 5 of this case. These labels attempted to comply with the letter of the law above mentioned by publishing the following sentence: “These goods are carefully prepared from selected stock and soaked in artesian well water.” The practice of preserving in leguminous vegetables a bright green color by the use of copper sulphate (blue vitriol) is illustrated by samples on the same shelves. In this connection it may be stated that some State laws require that vegetables containing copper salts shall be labeled with, or bear a paster stating, the amount of copper in each can.

Baking powder and baking powder chemicals.—Several illustrations of the frauds in this class of food adjuncts are given on the fifth shelf of this case. Whatever standards may be adopted for baking powders, the sale of an article whose composition is markedly different from that represented by the label must always be regarded as fraudulent. Insoluble matter, such as gypsum and talc, should not enter into the composition of substances of this nature.

Salad oils.—Olive, peanut, sunflower, and cotton-seed oils are all used extensively under their own names as foods, and no less than 15 oils are used as salad oil or to adulterate olive oil. The sale of a cheaper for a more expensive oil must be regarded as fraudulent.

CASE 4.

One of the important questions in connection with the preparation of foods is the extent to which foreign coloring matters may be legitimately employed. In most European countries it is forbidden to color artificially any substance which has a distinct color of its own; for instance, wine and fruit products must be sold without the addition of any foreign coloring matter. At the same time all countries permit the addition of some harmless colors, often specified by name, to foods and especially confections which are themselves colorless, but are ordinarily artificially colored. In some cases colors whose use with foods is permitted are not only specified by name, but the method by which such colors may be manufactured is given. This is done to exclude the use of coloring matters in whose preparation poisonous substances, such as arsenic, are employed. The fact that some of our common articles of food are often artificially colored at the present time is shown by the dyed fabrics displayed in the top of the three cases.

Heavy metals.—The use of tin cans as hermetically sealed receptacles for food has been of incalculable benefit in cheapening almost every article of food and prolonging the season of consumption for those which otherwise could be had only a comparatively short time. At the same time it must not be forgotten that an appreciable amount of heavy metals is often dissolved by the more acid varieties of food. The amount that is so dissolved depends on the age of the sample, the grade of tin plate employed, and the method by which the can was manufactured. Tin plate containing a large amount of lead is much more readily attacked than plate which is nearly free from lead. The plate which is manufactured by the oil process is more resistant to acids than that made by the acid process. Cans in whose manufacture zinc chlorid is used, often retain that salt between the seams, to be given up to the contents of the can. It is fortunate that the method of soldering with oil and resin is now usually employed.

Fruit products.—On the fourth shelf of this case is given an interesting collection of jelly and jam samples. The forms of adulteration to which this class of foods is subject are well indicated on the labels. This exhibit includes pure fruit preparations and products thickened with starch, sweetened with glucose and saccharin, preserved with salicylic acid, benzoic acid, etc., and artificially colored. A favorite practice is the preparation of jam from the refuse pulp of jelly manufacture. A sample of raspberry seeds found in "currant jam" is shown on the shelf above. The prices paid for these samples

are worthy of note. In this class of goods, as in others, adulteration is caused largely by the demand for cheap articles. A purchaser who pays no more for jelly than the glass which holds it and the sugar it is supposed to contain are worth should not expect a first-class article.

Little comment is necessary on the canned goods shown on shelf 5 of this case. The differences in value which attend the use of different sirups are not apparent to the eye. Even the quality of fruit employed can not always be determined by appearance alone. The branding of one variety with the name of another and labeling the goods of one locality as coming from another are always reprehensible. In this connection the recent court decisions forbidding certain Baltimore canners to label their goods as California products are of interest.

BEET-SUGAR INDUSTRY.

By E. E. EWELL.

The Department of Agriculture, through the Division of Chemistry, which July 1 was raised to the rank of a bureau, has been endeavoring to develop our domestic sugar industry for more than two decades. During the last ten years its efforts have been devoted principally to the development of the beet-sugar industry. Since 1897 this industry has grown rapidly and may now be considered to be on a permanent footing, as approximately \$25,000,000 are invested in the manufacture of beet sugar in this country. This does not include the large amount of capital invested in the growing of sugar beets.

The development and present condition of the industry in the United States is shown by a collection of statistical tables and photographs (pls. 1 to 40, referred to below), mounted in "wing frames" and designated as No. 5 on the diagram. (See frontispiece.) An effort was made in preparing the collection of photographs to illustrate as fully as possible all phases of the sugar industry from the production of the seed from which the beets are grown to the marketing of the sugar.

The modern sugar beet of high sugar content has been developed and its present high quality has been maintained by careful selection of the mother beets, from which seed is produced. At the beginning of the century the sugar beet contained only 5 to 6 per cent of sugar. The beets delivered to American factories in 1899 contained an average of 14.5 per cent of sugar, while the average of those grown in California was 15.9. Many single beets have been produced which contained more than 20 per cent of sugar, and the product of some entire fields has been found to contain nearly that amount. Until recently the seeds used for sugar-beet growing in the United States have been imported from the seed farms of Europe. The production of high-grade beet seed has now commenced in this country, and a

sample of seeds grown by the Utah Sugar Company, of Lehi, Utah, is exhibited in the case No. 6. (See Pl. I, frontispiece.) The production of sugar-beet seed has also been undertaken by the Spreckels Sugar Company, Spreckels, Cal., the Peninsular Sugar Refining Company, at Caro, Mich., and perhaps elsewhere.

Plates 5 to 13 of the exhibit illustrate the appearance of typical sugar beets and the methods used in this country for their cultivation, including the preparation of the land, the sowing of the seed, and other field operations, as well as the harvesting and delivering of the beets to the factory. Some sugar beets of great richness are shown in case No. 7. (See Pl. I, frontispiece.) They were taken from a lot of beets containing over 18 per cent of sugar.

GROWING AND MARKETING SUGAR BEETS.

In case No. 7 (see Pl. I, frontispiece) a map is exhibited which shows the areas in the United States probably suited to sugar-beet culture. This map was published with the reports of experiments with sugar beets made by the Division of Chemistry of the United States Department of Agriculture in 1897. Since that time twenty additional factories have been built and operated; eight more are in process of construction for the crop of 1901. All of these factories are located in or adjacent to the areas indicated on the map as probably suited to sugar-beet culture because of their favorable mean summer temperature. Subsequent experiments do not suggest, in general, that any material changes should be made in the areas indicated on this map as probably suited to sugar-beet culture. The Bureau of Chemistry is indebted to the Weather Bureau for valuable cooperation in the preparation of this map.

Unless the factory provides ample facilities for quickly weighing and unloading a large number of wagonloads of beets, the expense of delivering sugar beets to the factory is materially increased. Some of the methods employed for transferring sugar beets from wagons to cars or from wagons to the storage bins of the factory are shown on pls. 15 and 16 of the exhibit. The net method or some form of the tilting platform are very much used in various parts of the country. The net method is now very generally used on the Pacific coast. The net is placed in the bottom of the wagon and the beets loaded on top of it. When the wagon arrives at the factory or other unloading point, a rope is attached to the net, and by means of a horse or other power the beets are quickly transferred to the car or storage bin.

SUBSTANTIAL CONSTRUCTION OF AMERICAN FACTORIES.

The confidence of investors in the continued success of the industry is shown by the substantial character of the buildings which have been erected for the housing of the machinery used for the manufac-

ture of beet sugar in this country. Pls. 17 to 20 of the exhibit give an excellent idea of some of the typical forms of construction which have been adopted for this purpose.

STORAGE OF THE BEETS.

The factory must provide storage for many thousand tons of sugar beets. The device generally used is a shed or open bin with a trough-shaped floor along the bottom of which there extends a canal made of metal or masonry, which reaches from the storage place to the factory. This canal or sluiceway is covered with boards before the bins are filled with beets. When it is desired to transfer the beets from a given shed to the factory, these boards are pulled up one at a time and the beets allowed to fall into the canal, where they are caught up by a constantly flowing current of water and carried into the factory.

WASHING, WEIGHING, AND SLICING THE BEETS.

A large part of the adhering soil is removed during the hydraulic transportation of the beets, but in order to make the washing operation perfect the beets are passed through a special washing machine on their way to the slicing apparatus. In the modern factory the washed beets are weighed by an automatic scale, from which the superintendent or factory proprietor can learn at any moment the quantity of beets entering the factory during a given period, and compare it with the yield of sugar obtained and the richness of the beets as shown by the chemist's report. After the beets have been weighed they fall into the slicer, where they are cut into small slices having a V-shaped cross section by means of corrugated knives radially placed on a revolving disk. The storage sheds and the washing, weighing, and slicing machines are shown on pls. 21 and 22 of the exhibit.

EXTRACTION OF THE SUGAR FROM THE BEETS.

The extraction of the sugar from the sugar beet by means of pressure has given place to the more advantageous and more modern "diffusion process." This operation is conducted in a series of from 12 to 14 closed metal tanks connected by an elaborate system of pipes and valves, known in the sugar factory as the "diffusion battery." Each tank or "cell" in the battery holds one or more tons of beets, according to the capacity of the factory. The operation of the "battery" is as follows:

A cell is filled with slices of beets and the top door closed. Hot water is then admitted at the bottom until all of the space not occupied by the slices is filled with water. While the water is flowing into the first cell a second cell is being filled with beet slices, and as soon as this second cell is filled the conveyor which brings the slices to the "battery" is adjusted to deliver them into a third cell, and so

on, the current of slices being diverted to an empty cell as soon as one is filled. When the first cell is filled with liquid it is allowed to flow into cell No. 2, and through it into cells Nos. 3, 4, etc., as fast as they are filled with beets, and closed. The liquid flows from cell to cell through the system of piping referred to above, the current of water continuously entering cell No. 1. This is continued until all but two of the cells of the battery are filled. The manipulation is then varied by drawing liquid from the cell last filled with slices and liquid, into a measuring tank before the current of liquid is turned into the next cell of fresh slices. This portion of hot water has passed through ten or twelve portions of fresh beet slices, has approximately eight-tenths the density of the juice originally contained in the beets, and is called "diffusion juice." The sugar is extracted from the beets partially by a process of displacement of the juice by hot water, but largely by the process of diffusion, the sugar diffusing from the slices into the liquid which surrounds them. The beet slices contained in the cell which was first filled have now been washed with ten or twelve successive portions of water, and contain less than one-half of 1 per cent of sugar. This cell is therefore emptied in order to make room for more fresh slices. The process is continuous. Each time a cell is filled with fresh beet slices and juice, and a portion of diffusion juice drawn off, the cell at the opposite end of the line contains exhausted slices and is emptied.

The cells of a "diffusion battery" are sometimes arranged in a straight line and sometimes in a circle. The appearance and construction of different forms of batteries are shown on pls. 23 to 26 of the exhibit.

PURIFICATION OF THE JUICE.

The "diffusion juice" is submitted to an elaborate process of purification before it is in a condition suitable for evaporation for the recovery of the sugar contained in it. Lime and carbon dioxide (carbonic-acid gas) are the agents principally employed for this purpose. They are both obtained from a lime kiln, which is kept continuously in operation (pl. 27 of the exhibit). The kiln is charged with coke and limestone at the top and the burnt lime is withdrawn at the bottom. Carbon dioxide is formed in large quantities in the kiln, both by the combustion of the coke and by the decomposition of the limestone. It is withdrawn at the top of the kiln by means of pumps, and forced through the juice contained in the "carbonatation tanks" (see pls. 27 and 29 of the exhibit). The process of purification includes five filtrations and two successive treatments with lime and carbon dioxide. The juice coming from the battery is passed through coarse filters for the purpose of removing small fragments of beet pulp. It is then treated with a large excess of lime (2 to 3 per cent of the weight of the beets worked), and carbon dioxide is then forced through the hot juice until

the lime which has not already been rendered insoluble by combination with organic acids and other impurities in the juice is precipitated as carbonate of lime. The "carbonated" juice is very turbid, and is pumped through filter presses (pls. 27-29 of the exhibit), which remove the insoluble matter suspended in it. This insoluble matter consists of carbonate of lime, lime compounds formed with the impurities contained in the juice, proteid bodies rendered insoluble by the heat, etc. The clear juice is again treated with lime and carbon dioxide, and again filtered. The clear juice coming from the second "carbonatation" is then bleached with sulphurous acid, applied in the form of the fumes of burning sulphur. The bleached juice, after being again filtered, is ready for the evaporator.

EVAPORATION OF THE JUICE TO SIRUP.

The juice, after having been purified by the method described, is evaporated to a sirup containing about 50 per cent of water and 50 per cent of solid matter. The solid matter is made up of 40 to 45 per cent of sugar and 5 to 10 per cent of organic and inorganic substances other than sugar, which, to a greater or less extent, prevent the crystallization of the sugar.

In the modern sugar factories the juice is evaporated in multiple-effect vacuum evaporators heated by steam. These evaporators consist of from two to four closed evaporating pans which are heated by steam which circulates through copper pipes arranged in the lower part of the first pan. They are called "multiple effect" because more than one effect of a given portion of steam is obtained. For example, exhausted steam from the pumps and engines or steam drawn directly from the boilers is used to heat the first pan. The steam generated by the boiling juice in this pan is collected, and used to heat the second pan. The vapor of the second pan is in turn used to heat the third pan. In the case of a quadruple-effect evaporator the vapor in the third pan is used to heat a fourth pan. The vapor from the last pan is conveyed to a condensing apparatus in which it meets a spray of cold water, which condenses the vapor to form water.

Air leaking into the apparatus and noncondensable vapor are removed from the condenser by means of a vacuum pump, which is kept constantly in action. The juice in the first pan boils at approximately the atmospheric pressure. The pressure in the following pans is successively reduced from pan to pan, the liquid in the last pan boiling in as nearly a complete vacuum as is practicable. Evaporators of this type are called "double," "triple," or "quadruple effects" accordingly as there are two, three, or four pans. The operation of the evaporators is continuous. The juice is pumped into the first pan while the finished sirup is pumped out of the last pan in a continuous stream. The collection of photographs already referred to contains sectional and exterior views of two types of evaporators (pls. 30 and 31).

It is the practice in most modern factories to filter the sirup after it comes from the evaporator, as more or less insoluble matter separates from the juice during the process of evaporation.

GRANULATION OF THE SIRUP.

The filtered sirup is granulated in a vacuum apparatus which the sugar maker calls a "strike-pan." The appearance of the granulating pan is shown on pl. 32 of the exhibit.

The operation of the vacuum pan is one of several processes conducted in the beet-sugar factory that requires a great amount of skill. The pan is heated by several tiers of copper coils so connected that steam can be admitted to any one of them at the will of the operator. Sirup is drawn into the pan and concentrated until the pan is from one-fourth to one-third filled with a liquid of such density that when a fresh charge of cool sirup enters a large number of small crystals of sugar is formed.

The boiling is then continued in such a manner that these crystals continue to grow in size until the entire pan is filled with a thick mass of crystals of sugar and molasses. The number of crystals formed at the beginning of the boiling determines the size of the crystals, or the "grain" of the finished product. When the operation is skillfully conducted no additional crystals, or "false grain," as it is called, are allowed to form after the original quantity of "grain" which the sugar boiler considers the proper amount has been formed. The coarse and fine granulated sugars found in the market are due to the manner in which the boiling is conducted and not to any subsequent crushing or grinding operation.

SEPARATION OF THE SUGAR FROM THE MOLASSES.

The finished product of the vacuum pan is known in the sugar house as "masse-cuite," and is separated into molasses and sugar by means of centrifugal machines. These machines consist essentially of a cylinder having walls of fine wire cloth and attached to a vertical shaft in such a way that they can be made to revolve at a speed of from 1,200 to 1,500 revolutions per minute. Small portions of the "masse-cuite" are placed in these machines, which are then rotated at the rapid rate named. The centrifugal force causes the molasses to pass through the mass of sugar and the screen, while the crystals are retained thereon. Any adhering molasses is removed from the crystals of sugar by means of a spray of water to which a small portion of ultramarine blue (a harmless coloring matter) is added to correct the yellow tint which the sugar would otherwise have. The construction and outer appearance of the centrifugal machines may be seen by inspecting the photographs on pl. 33 of the exhibit.

DRYING AND PACKING THE SUGAR.

The sugar as it comes from the centrifugal machine is moist and must first be passed through the "granulator" before it is ready for

packing. One form of "granulator" is shown on pl. 33 of the exhibit. It usually consists of a revolving drum through which the sugar is made to pass in contact with a current of hot air. The sugar is packed for the market either in barrels or in burlap sacks lined with muslin. The packing room and a train load of sugar are shown on pl. 34 of the exhibit.

EXHIBIT OF FACTORY PRODUCTS.

The sugars and intermediate products made in a number of American beet-sugar factories are exhibited in case No. 6 (see Pl. I, frontispiece). The final product of most of the factories is a high-grade granulated sugar suitable for table use. In two or three cases, however, raw sugar is made which must first go to the refinery and be subjected to a refining process before it is suitable for consumption.

It is quite generally believed that granulated sugar made from sugar beets does not possess the sweetening power of the corresponding product made from sugar cane. When the granulated sugar from the two sources has in each case been skillfully made by modern processes, the product, for all practical purposes, is precisely the same, whether it comes from sugar beets or from sugar cane. It consists of more than 99.5 per cent of pure cane sugar mixed with less than one-half per cent of other materials which consist of small amounts of moisture, mineral matter, and other substances which do not essentially alter the character of the product. Chemists have never been able to detect any difference between the thoroughly purified sugars from the two sources, and this sugar, when separated in pure form, is called cane sugar, whether it comes from beets or from sugar cane, because it was originally found in the latter and manufactured therefrom for commercial purposes. Therefore, as the commercial products are rendered more and more pure by the use of good manufacturing processes, the difficulty of telling the source from which the sugar is obtained (beet root or sugar cane) becomes greater and greater. Poorly made granulated sugars obtained from beet roots possess to a greater or less extent a characteristic odor which can best be observed by placing the sugar in a bottle or other receptacle that can be tightly closed and noting the odor of the air surrounding the sugar in the bottle immediately after the cork has been removed and after the sugar has been closed up in the bottle for one or more days. When proper skill and the most improved processes are used for the manufacture of sugar from beets, this characteristic of the product becomes much less and, for all practical purposes, disappears. Therefore, statements that the sweetening power of sugar made from sugar beets is essentially different from that made from sugar cane are erroneous. It is of course true that while many of the raw sugars and other unrefined products from sugar cane are very palatable this is not the case with the unrefined products of the beet-sugar factory.

IMPORTANT BY-PRODUCTS.

Samples of the important by-products, beet pulp both in a dry and in a fresh condition, and the lime-cake fertilizer, can also be found in cases Nos. 6 and 7. (See Pl. I, frontispiece.) The lime-cake fertilizer is prepared by drying and grinding the filter-press cake, which consists of the insoluble matter removed from the carbonated juice by filtration. The sample exhibited has the following chemical composition:

Composition of the lime-cake fertilizer sent by American Beet Sugar Company, Chino, Cal.

	Per cent.
Phosphoric acid (P_2O_5)	0.35
Potash (K_2O)	2.30
Lime (CaO)	41.65
Carbonic acid	23.20
Nitrogen	.17
Equivalent to ammonia (NH_3)	.21

The most important by-product of the beet-sugar industry, particularly from the standpoint of the farmer, is the exhausted beet pulp, which forms a most excellent stock food either in a moist or in a dried condition. The pulp as it comes from the battery contains approximately 95 per cent of water, but a considerable part of this is removed by passing the pulp through some form of continuous press (pl. 35 of the exhibit). The pressed pulp contains 85 to 90 per cent of water. A load of pressed pulp therefore contains about twice as much food material as does the same weight of the unpressed pulp. Recently some of the factories in this country have been equipped for rendering the pulp still more suitable for transportation by drying it in especially constructed drying plants until only about 10 per cent of moisture remains in it. Samples of both fresh and dried pulp are shown in cases Nos. 6 and 7. (See Pl. I, frontispiece). The American farmers have been slow to utilize the exhausted beet pulp for feeding purposes. Its use, however, is being extended every year, and doubtless within a short time none of it will be allowed to go to waste.

The sample of dried sugar-beet pulp exhibited was made at the factory of the Bay City Sugar Company of Bay City, Mich. Its chemical composition was as follows:

Composition of dried sugar-beet pulp.

(1) Results of analyses made to determine food value:	Per cent.
Moisture	9.56
Fat	.99
Protein (nitrogen $\times$ 6.25)	7.13
Fiber	18.30
Ash	3.73
Digestible carbohydrates, etc., by difference	60.29
(2) Results of analyses made to determine fertilizer value:	
Nitrogen	1.14
Equal to ammonia	1.38
Phosphoric acid (P_2O_5)	.34
Potash (K_2O)	.19

For the average composition of pressed and dried beet pulp in comparison with certain other feeding materials in common use, see the Yearbooks of the Department of Agriculture, 1898, pp. 213-220. and 1900, p. 752.

The factory at Leavitt, Nebr., may be mentioned as one of those at which special attention has been given to the utilization of the exhausted beet pulp for stock feeding. Several views taken on the farm of the Standard Cattle Company, Ames, Nebr., which uses pulp from the factory at Leavitt, are shown on pls. 35 and 36 of the exhibit.

The appliances used at the factory of the California and Hawaiian Sugar Refining Company's factory at Crockett, Cal., for the transportation of sugar-beet pulp from the factory to the silos are shown on the large photograph exhibited in case No. 7. (See Pl. I, frontispiece).

POWER AND LIGHTING.

Pls. 37 and 38 of exhibit show typical views in the boiler and engine rooms and the electric-lighting plants of American beet-sugar factories. The fuel employed is generally coal, with the exception of California, where crude petroleum is very largely used.

THE BEET-SUGAR MANUFACTURER'S DEBT TO SCIENCE.

The present condition of the beet-sugar industry is the result of the application of scientific methods in the field and in the factory. While other sciences have contributed largely analytical chemistry has been the most important factor in the development of the modern high grade sugar beet and in perfecting the methods of manufacture. Case 7 (see Pl. I, frontispiece) has been utilized for exhibiting some of the best methods and appliances for the analysis of the sugar beets and the sugar house products for the control of manufacturing processes. The usefulness of the analytical laboratory begins with the production of the sugar-beet seed. All of the larger seed farms in Europe, and those which are now being established in this country, are provided with laboratories equipped for the analysis of the "mother" beets in order that only those containing a high percentage of sugar and a very pure juice may be replanted for seed production. The methods employed for the analysis of beets which are to be selected for seed production must be rapid and inexpensive in order that a large number of beets, often hundreds or thousands, may be analyzed daily without too great an expenditure. The determination of sugar by means of a polariscope is now the means generally employed for this purpose. Various methods have been devised for removing a small portion of the beet and extracting the sugar therefrom, preparatory to determining its amount in the polariscope; a small portion of each beet can be taken without lessening its value for seed production.

EXHIBIT OF METHODS AND APPARATUS FOR THE ANALYSIS OF SUGAR BEETS.

One of the most rapid processes (shown in the case mentioned) consists in cutting out from each beet a small cylinder with an apparatus similar to an apple corer. From this cylinder a portion is cut with the Pellet parallel-bladed knife, the knives being so adjusted that the piece removed will weigh an aliquot part of the normal weight of sample arbitrarily adopted for the polariscope used. This sample portion is reduced to a pulp by the Hanriot machine and washed into a graduated flask. A small quantity of lead acetate solution is then added and the mixture diluted with water to a definite volume, thoroughly mixed by shaking and filtered. A tube, closed with glass disks at each end, is filled with the clear filtrate and placed in the polariscope; the percentage of sugar is then read directly upon the scale. This method sacrifices accuracy to some extent, but gains in rapidity by eliminating the use of a balance for determining the quantity of beet pulp to be used for each analysis.

There is also exhibited the Keil and Dolle boring rasp, which can be used either for sampling beets for seed selection or for sampling the beets at the factory receiving laboratory. The Pellet conical rasp, suitable only for the latter purpose, is also shown. Small apparatus for expressing juice from beet pulp for the analysis of beet juice for the purpose of seed selection also form a part of the exhibit.

Two polariscopes are shown: a small one for the analysis of sugar beets for seed selection, and a larger one of one of the latest improved models, with an incandescent electric lamp for illumination, used when greater accuracy is required. An important accessory of the polariscope is the Pellet continuous tube, which greatly increases the number of sugar solutions which can be examined in the polariscope in a given time. Both the methods and the instruments for the polariscopic determination of sugars have been greatly improved during recent years, until at the present time results of the highest accuracy can be obtained when the necessary precautions are observed. Recently the influence of temperature on sugar determinations by means of the polariscope has been carefully worked out by several investigators, and tables of factors, prepared by means of which corrections may readily be made for the slight errors necessarily introduced by the varying temperature of the laboratory.¹

For the encouragement of farmers to produce the richest beets possible in a given region, it has long been the practice in Europe and in this country to vary the price paid the farmer according to the percentage of sugar in the beets. Formerly it was the practice in Europe

¹See article by Harvey W. Wiley, On the Influence of Temperature on the Specific Rotation of Sucrose and Method of Correcting Readings of Compensating Polariscopes therefor. *Journal of the American Chemical Society*, 1891, 21, 568-596.

to simply determine the density of the juice of the beet by means of the hydrometer, inasmuch as it is a fair indication of the percentage of sugar contained therein. With the improvement of analytical processes, both in accuracy and in rapidity, it has now become customary to make an actual determination of the percentage of sugar contained in each lot of beets delivered at the factory. Two types of methods are in use for this purpose:

(1) The determination of the sugar contained in the juice expressed from the beet and the calculation of the percentage of sugar contained in the beet by means of an arbitrary factor.

(2) The direct determination of the sugar in the beet by means of extraction with alcohol, hot water, or, more recently, with cold water.

The indirect method involves the use of an arbitrary factor which has frequently given rise to controversy. The percentage of juice contained in the beet varies with the conditions under which they were grown and with the time and manner of their storage after removal from the ground. It is therefore a difficult matter to decide upon the proper factor for the calculation of the percentage of sugar in the beet in each individual case. It is usually not practicable in the receiving laboratory of the factory, which is a very busy place and a very expensive adjunct of the factory, to do more than adopt an average factor and apply the same to all beets received.

The direct methods of analysis have, however, been recently so greatly improved in point of rapidity that it seems that factory proprietors may now consider their adoption for the analysis of beets in their receiving laboratories. For their successful application, however, it should be remembered that the working rooms and the laboratories should be provided with a great abundance of apparatus of the most improved pattern as well as ample room for conducting the various analytical operations. No economy will be found in limiting either space or equipment in the receiving laboratory. The exhibit of apparatus in the case named contains several devices which will be found useful for rapidly and accurately executing a large number of beet analyses.

In addition to a receiving laboratory where the beets are analyzed as they are delivered by the farmers, the beet-sugar factory has a laboratory for the analysis of the exhausted slices, juices, sirups, massecuites, and other products of the factory. These analyses are necessary to enable the proprietor or factory superintendent to determine whether all of the different manufacturing processes are being conducted in a proper manner.

The laboratory of an American sugar factory is shown on pl. 40 of the collection of photographs.

In this connection it is proper that acknowledgment should be made of the samples of sugar-house products, photographs, and other materials contributed by the manufacturers of beet sugar in various

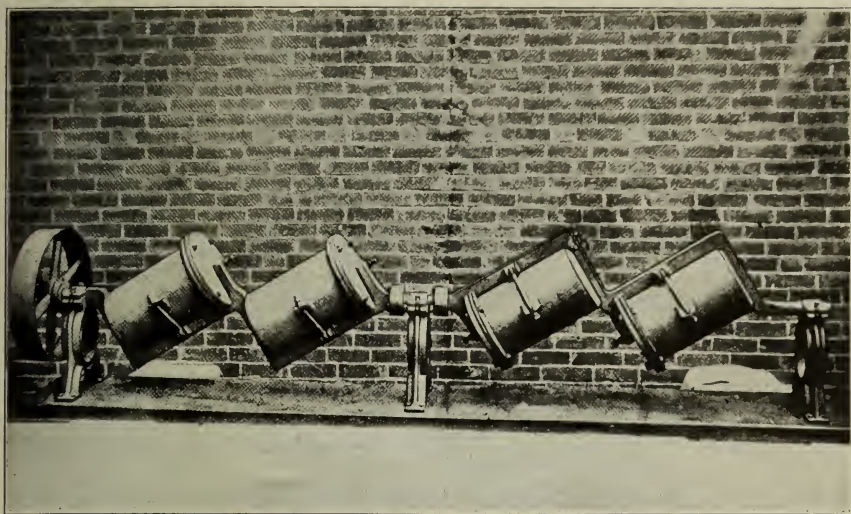


FIG. 1.—ABRASION MACHINE.

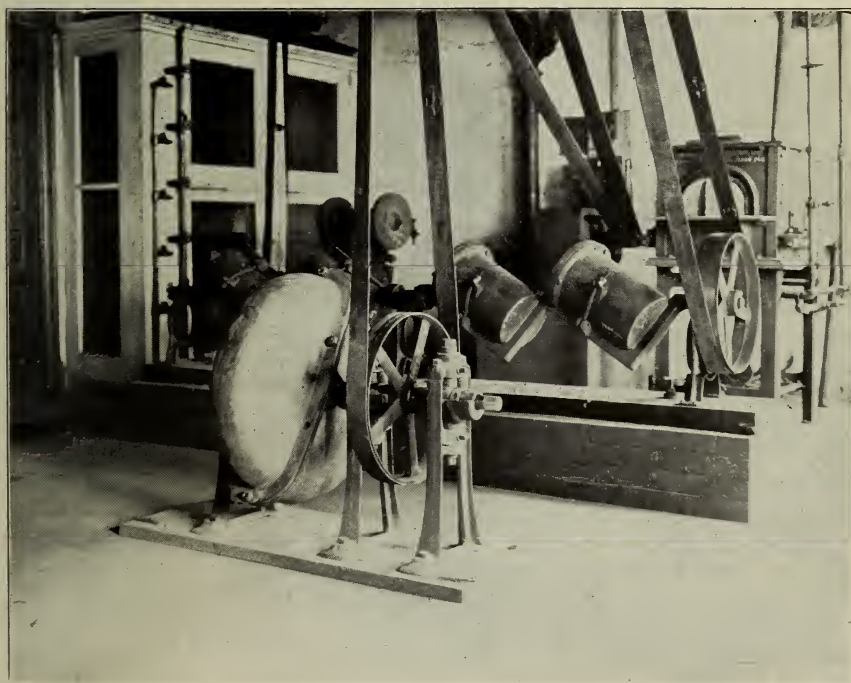


FIG. 2.—BALL MILL.

parts of the country. Without this cooperation on the part of the factory proprietors this exhibit would have been impossible.

EXHIBIT OF ROAD-MATERIAL LABORATORY.

By LOGAN WALLER PAGE.

The Road-Material Laboratory was established in October last for testing road materials free of charge for any citizen of the United States. As most of the machinery necessary for this work had to be especially designed, the actual testing of road materials was not begun until December.

The short time which has intervened since then, together with the lack of available funds, has made it impossible to do more than install a part of the equipment necessary for the testing of rock and gravel. The present exhibit is consequently far from complete. In order to make an understanding of the exhibit as clear as possible, a brief description will be given of the entire process of testing rock and gravel at the present time.

When a request is received at the laboratory for making a test, a blank form containing a number of questions as to the exact location from which the material comes, by whom owned, and the amount available is sent, together with a tag envelope and instructions for selecting and shipping. In the case of rock, as soon as a sample is received at the laboratory, a hand specimen about 4 by 6 by 1 inches in dimensions is properly dressed and put aside for reference. Examples of such hand specimens can be seen in exhibit case No. 8. (See Pl. I, frontispiece).

ABRASION TEST.

The first test made on rock is the abrasion test, for determining the relative resistance to wear. This test is made by placing 5 kilograms (11 pounds) of rock of the usual size employed in road making (3 to 6 cm) in one of the cylinders of the abrasion machine. (See Pl. II, fig. 1.) These cylinders are 34 cm in depth and 20 cm in diameter, and are arranged on a shaft at an angle of 30° to the axis or rotation of the shaft. The cover of the cylinder is bolted on, and the cylinder is allowed to rotate for five hours, making 10,000 revolutions. Each revolution of the cylinder throws the fragments of rock from one end to the other twice, which causes them to grind and pound against one another and against the walls of the cylinder. The contents of the cylinder, after 10,000 revolutions, is brushed into a basin and the resulting fine detritus is sifted into several sizes (shown in the exhibit under the head of abrasion-test results) with an automatic sifter, a photograph of which is shown in the exhibit case. From the dust worn off two coefficients of wear and the percentage of wear are computed.

The Department coefficient of wear is obtained by subtracting 4,000 from the weight of the unabraded fragments of rock and dividing the difference by 10. This allows a possible range in results from zero to 100, i. e., if 1,000 grams of the material is abraded from the original 5 kilograms the result will be zero, and the material is considered unfit for road making. If no dust is worn from the original 5 kilograms the coefficient will be 100.

The French coefficient of wear is obtained by the following formula:

$$\text{Coefficient of wear} = 20 \times \frac{20}{w} = \frac{400}{w}$$

where "w" is the weight in grams of dust under 0.16 mm. (one-sixteenth of an inch) in size, obtained per kilogram (2.2 pounds) of rock used, 20 being the standard of excellence.

The percentage result includes all sizes of material worn from the original 5 kilograms. The results of 8 abrasion tests can be seen in the exhibit case.

This test was designed by M. Deval, and was first exhibited in Paris in 1878. It is, as far as the writer has been able to ascertain, the first machine especially constructed for testing road materials.

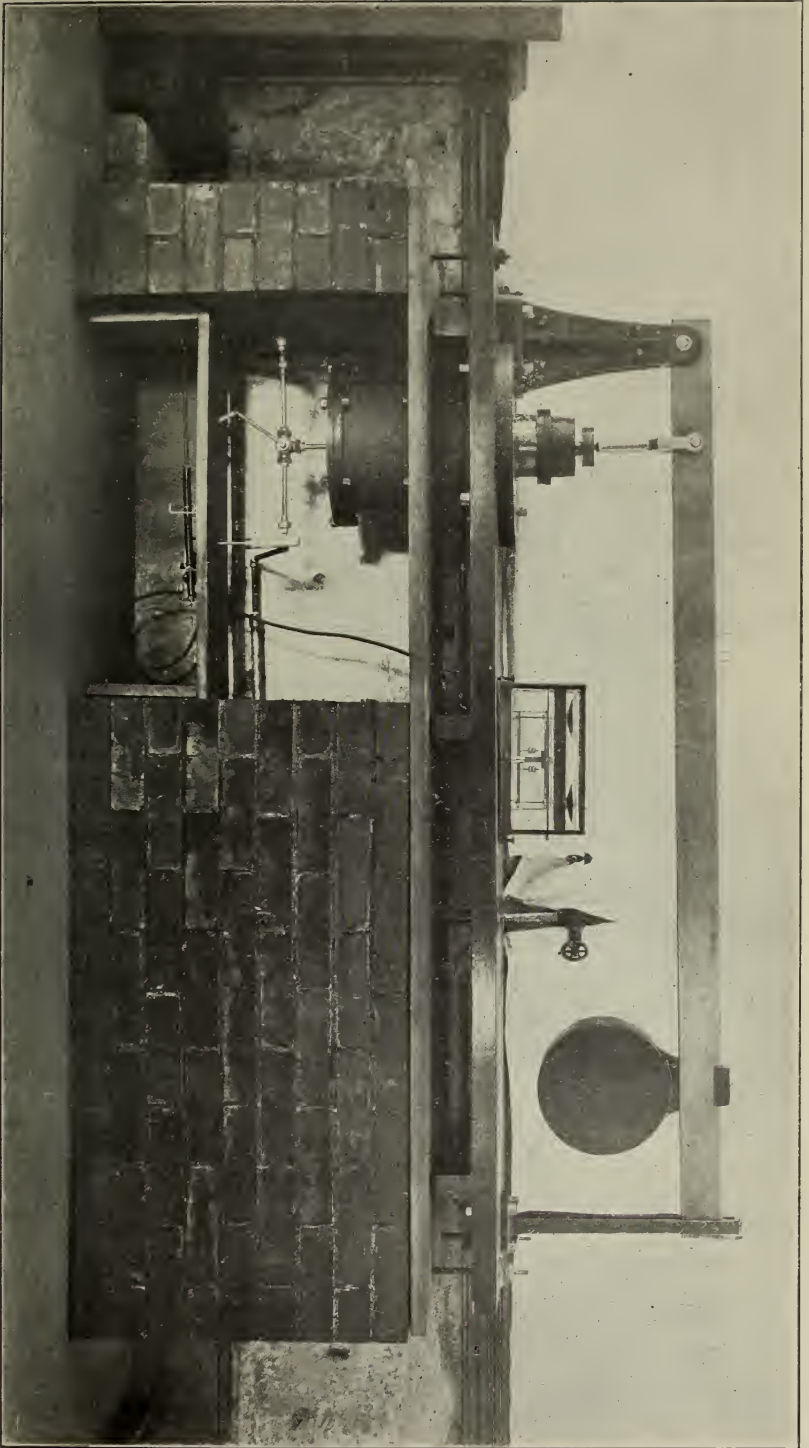
ABSORPTIVENESS AND DENSITY OF ROCK.

As soon as the abrasion test is completed one of the smoothly worn fragments, from 20 to 40 grams in weight, is used for determining the absorptiveness of the sample, which is done in the following manner: This smoothly worn stone is weighed in air after it has been brought to a constant weight in a hot-air bath. It is then immersed in water and immediately reweighed in water. After ninety-six hours of immersion it is again weighed in water.¹ The absorption is obtained by the following formula:

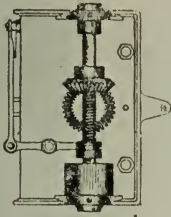
Number of pounds of water absorbed by a cubic foot of rock $= \frac{C-B}{A-B} \times 62.5$, in which "A" is equal to the weight in air, "B" the weight immediately after immersion in water, "C" the weight after absorption for ninety-six hours, and 62.5 the weight of a cubic foot of water.

This method has no particular advantages over others except that it requires less manipulation and time and the result is a little more practical. It is not intended to give the porosity of rock, but merely to obtain from a small specimen the number of pounds of water absorbed by a cubic foot of rock in ninety-six hours. It can be readily seen that the specific gravity and weight of a cubic foot of the rock can be determined from the above data. Two such trials are always made for each sample, and three or more when necessary.

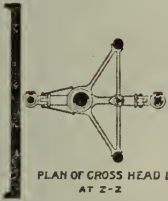
¹ It was found by experiment with a number of stones that absorption practically ceases after sixty-two hours, so ninety-six hours was given as a safe allowance.



BRIQUETTE MACHINE.

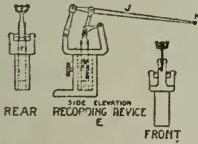


PLAN OF TOP



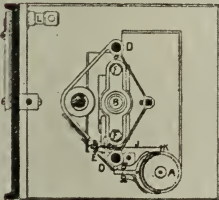
PLAN OF CROSS HEAD I
AT Z-Z

IMPACT
TESTING MACHINE

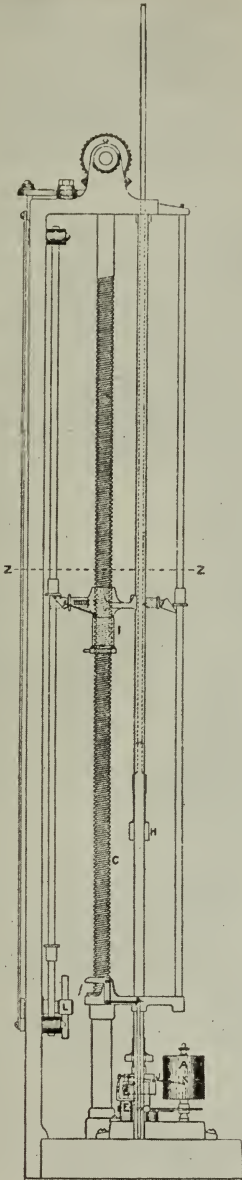


REAR
SIDE ELEVATION
RECORDING DEVICE
E

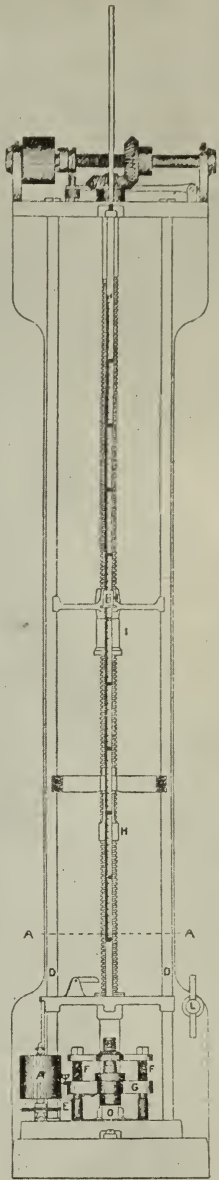
FRONT



SECTIONAL PLAN A-A



SIDE ELEVATION



FRONT ELEVATION

IMPACT MACHINE.

During the coming year it is intended to determine the density of rock by careful measurement with a micrometer screw, and the porosity by means of a vacuum pump and hot water, which will be much more accurate than the methods at present employed.

CEMENTATION TEST.

The next test in order is the cementation test, which is intended to determine the important cementation or cohesive property of rock, which, when possessed in a high degree, gives an impervious shell to the surface of a road.

This property is determined by taking 500 grams of the sample broken to pass through a screen of 6 mm mesh, but not through a 0.5 mm mesh. The material thus broken is placed in a ball mill (Pl. II, fig. 2) and reduced to a powder that will pass through a 0.25 ($\frac{1}{16}$ inch) mm mesh. This dust is made into briquettes of circular section, 25 mm (0.98 inch) in diameter and 25 mm in height, by mixing the dust with distilled water (about 4 cc) on a glass plate and placing it in a steel die of the proper dimensions. This die has a closely fitting steel plug which is inserted over the dust, and the dust is subjected to a pressure of 100 kilograms per square centimeter (1,422 pounds per square inch) with a machine especially in designed for the purpose, which gives perfectly uniform conditions in making each briquette. (See Pl. III, also photograph in exhibit case.) The weight of the dust varies with the density and compressibility of the rock, but generally it requires about 23 grams (0.8 of an ounce) of dust to make a briquette of the above dimensions. These briquettes are kept in a hot-air bath until their weight becomes constant, and are then placed in a desiccator and allowed to cool, after which they are broken by impact.

The machine especially designed for this test (see Pl. IV) consists of a 1 kilogram (2.2 pound) hammer (H), arranged like the hammer of a pile driver on two vertical guide rods (D). The hammer is raised by a screw (C) and dropped automatically from any desired height. It falls on the flat end plunger (B) of 1 kilogram weight, which is pressed upon the briquette (O) by two light spiral springs held on the guide rods (F). The plunger (B) is bolted to the crosshead (G), which is held by two vertical rods (F). A small lever (J), carrying a brass pencil (K) at its free end, is connected with the side of the crosshead by a link motion, arranged so that it gives a vertical movement to the pencil six times as great as the movement of the crosshead. The pencil is pressed against the drum (A), and its movement is recorded on a slip of silicated paper fastened thereto. The drum is moved automatically through a small angle at each stroke of the hammer. In this way a record is obtained of the movement of the hammer after each blow. The standard fall of the hammer for a test is 1 cm. (0.39 inch). This blow is repeated until the bond of cementation of

the material is destroyed. The final blow is easily ascertained, for when the hammer falls on the plunger, if the material beneath it withstands the blow, the plunger rebounds; if not, the plunger stays at the point to which it is driven, which is recorded on the slip of paper. The automatic record thus obtained from each briquette is filed for future reference. The number of blows required to break the bond of cementation, as described above, is taken as representing the cementing value of the rock, and is so used in comparing this property in road materials. Five briquettes are tested from each sample and their average taken for the result. Briquettes can be seen in the exhibit case before and after they have been subjected to this test; also records of results.

RECEMENTATION TEST.

The fragments of the five briquettes that are broken are again passed through a 0.25 mm. screen, made into briquettes, and broken again to determine the recementation value. This property is in a way more important than the cementing value, for the dust on a road surface is continually being recemented. With most materials about 50 per cent of the cementing value is lost on recementation, but with a few it increases.

OTHER TESTS.

Besides the tests enumerated above, the character of each sample is studied, and each rock is properly classified according to its mineralogical composition.

During the present fiscal year a number of new tests will be taken up. Among the most important is a test for determining the toughness of rock, which will be made in the following manner:

A machine similar in all respects to the present machine used for breaking the rock-dust briquettes will be used, except that it will have a spherical end plunger instead of the present flat one, and cubes of rock will be substituted for the briquette. The test will consist in a drop of the hammer of 1 cm. for the first blow, and an increased drop of 1 cm. for each succeeding blow, until the cube is destroyed. The entire energy of each blow is thus concentrated at the center of the upper surface of the cube, splitting the cube from this point after the elastic limit is reached. The number of blows required to destroy a cube will be used to indicate the toughness of a rock. It can be seen that this treatment approximates very closely the blows of horses' feet and the wheels of vehicles to which road materials are subjected.

TESTS ON GRAVEL.

The only test made on gravel at the present time is a determination of its cementing value, which is made by grinding 500 grams of a

sample in the ball mill referred to above, making it into briquettes, and testing them in the same way in which the rock samples are tested.

Apparatus is now in preparation for making mechanical analyses of gravel, which will doubtless prove of much value. These analyses will consist in separating the natural sizes of the gravel by precipitation in water. The present plan is to separate it into the following sizes: Between 2 and 1 mm., 1.5 mm.; 0.5 to 0.25 mm.; 0.25 to 0.1 mm.; 0.1 to 0.05 mm.; 0.05 to 0.01 mm.; 0.01 to 0.005 mm.; 0.005 to 0.0001 mm.

The testing of paving brick will also be taken up during the present fiscal year.

Those desiring road materials tested must apply to Road-Material Laboratory, Bureau of Chemistry, Department of Agriculture, Washington, D. C.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY.

THE INFLUENCE OF ENVIRONMENT

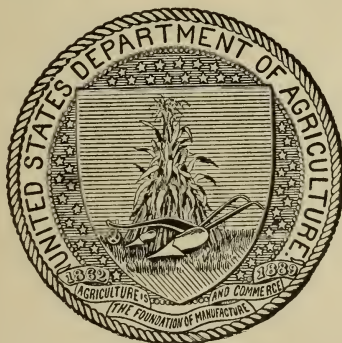
UPON THE

COMPOSITION OF THE SUGAR BEET.

BY

HARVEY W. WILEY,
CHIEF OF BUREAU,

IN COLLABORATION WITH THE WEATHER BUREAU AND THE AGRICULTURAL
EXPERIMENT STATIONS OF INDIANA, IOWA, KENTUCKY, MICHIGAN,
NEW YORK, CORNELL UNIVERSITY, NORTH CAROLINA,
UTAH, AND WISCONSIN.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1901.

LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., July 30, 1901.

SIR: I have the honor to submit herewith for your inspection and approval manuscript and graphic charts showing the results of the study by the Division of Chemistry, in collaboration with a number of the experiment stations and with the Weather Bureau, of effect of environment upon the chemical composition of the sugar beet. I recommend that this manuscript be printed as Bulletin No. 64, Bureau of Chemistry.

Respectfully,

H. W. WILEY, *Chief of Bureau.*

Hon. JAMES WILSON, *Secretary.*

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THE INFLUENCE OF ENVIRONMENT UPON THE COMPOSITION OF THE SUGAR BEET.

ORGANIZATION OF COLLABORATIVE WORK.

For more than a quarter of a century the Division of Chemistry of the Department of Agriculture has been studying the effect of environment upon the composition of the sugar beet in so far as its content of sugar is concerned. The present bulletin will be devoted principally to the study of climatic influences, reserving for future monographs the rôles played by the soil and applied fertilizers.

The early studies of this division emphasized the fact, already pointed out by European investigators, that beets grown in more northern latitudes show a higher content of sugar than when grown farther south. So strongly were these facts brought out by our own investigations that it was deemed advisable to limit, or at least suggest the limitation of, the growth of the sugar beet for commercial purposes to the more northern portions of our country. When, later on, the development of the arid regions showed the possibility of the production of beets of high grade, a totally different problem was presented for consideration—a problem which had never been entered upon by investigators of agricultural science. It is evident that the factors which are dominant in irrigated areas are very different from those which determine the character of the product in areas where the rainfall is usually sufficient for the production of the crop. In the present bulletin our studies have been confined to the usual climatic conditions prevalent throughout the greater part of the United States devoted to agriculture. In order, however, to include at least some idea of the nature of the problem in irrigated regions, one station, namely, that of Utah, was invited to collaborate in the work. The Bureau¹ is greatly indebted to the directors of the agricultural experiment stations who consented to collaborate in the work, not only for the heartiness and value of their cooperation, but especially for the reason that this collaboration was given without adequate compensation. Inasmuch as the funds available for the investigation were very limited and scarcely more than sufficient to conduct the chemical work at the Department

¹ On July 1, 1901, the Division of Chemistry became a Bureau.

of Agriculture, it would have been impossible to have carried on the work without the generosity of the collaborating stations. The following agricultural stations were invited to cooperate in the work and all accepted, namely: Indiana, Iowa, Kentucky, Michigan, New York, North Carolina, Utah, and Wisconsin. In New York both the State station at Geneva and the Cornell station at Ithaca were invited to collaborate.

The following letters were sent to the directors of the several stations named on April 4 and 17, 1900:

APRIL 4, 1900.

DEAR SIR: I have just received from Mr. Maurus Deutsch, one of the progressive sugar-beet seed growers of Austria, a small quantity of the very highest grade of sugar-beet seed, of three different varieties of the Austrian Kleinwanzlebener. I should like very much to have this seed planted with the greatest care, cultivated in the highest style of the art, and analyzed at the period of full maturity.

If you can use a small quantity of this seed, say enough to plant an eighth or a fourth of an acre, I shall be very glad to send it on to you, together with a full description of the names, etc.

Please let me hear from you at your earliest convenience in regard to this matter.

Very truly, yours,

H. W. WILEY, *Chemist*.

APRIL 17, 1900.

DEAR SIR: I take pleasure in sending by separate mail the high-grade Austrian beet seed of which I wrote you a short time ago.

The purpose I have in view in asking your collaboration in this matter is to make a careful study of the influence of climate on the character of the beets grown. To this end the seed has been distributed over a wide range of meteorological conditions, and the result of the study will be of the greatest interest.

For this reason I have requested your aid, and would ask you to take every possible care in the growth of these beets. I especially want the seed planted in sufficient quantities to insure a perfect stand. These seeds have not been tested by the division here, because I did not want to wait until that was done, and therefore I would ask you to plant them at the rate of at least 30 pounds per acre, under the most favorable conditions possible. I would also ask you to attend carefully to the cultivation of the beets, and to keep a record of the cultivation data and the meteorological conditions which prevail during the growing season.

For analytical purposes I would be glad if you would send here, from time to time, representative samples of the beets. Mailing facilities will of course be granted you for this purpose. If you have not already the use of the frank of the Department for this purpose, please inform me at the time of harvest and I will send special franks for the transmission of samples through the mails, and also the directions for securing the representative samples desired.

I would like also to have the analyses made at your own laboratory, if you have time to do so.

I thank you most heartily for your consent to enter into this collaborative work, which I trust may prove of advantage to your station.

Very truly, yours,

H. W. WILEY, *Chemist*.

To each of the stations collaborating the requisite quantity of the Austrian Special Kleinwanzlebener beet seed was sent. There was no special reason for the selection of this particular variety of seed other than that it was produced from mother beets which had been selected by analysis on account of their high sugar content. The tendency of such seeds would therefore be to produce beets of uniformly high grade. It is evident that any variation in the quality of the beets grown in different localities from the same seed must be due to the environment, namely, soil, fertilization, culture, and meteorological conditions.

EXPERIMENTS CONDUCTED AT WASHINGTON, D. C.

A plot on the agricultural farm situated on an island, or reclaimed lands, of the Potomac River, lying south of Aqueduct Bridge, was also planted with the Austrian Special Kleinwanzlebener beet seed and subjected to the ordinary careful cultivation necessary for the production of high-grade beets. These lands, being sufficiently fertile, received no fertilizer of any kind. The soil was formed of débris taken from the Potomac River by dredging machines, and therefore it has no geological characteristics. It is a mixture of silt, sand, and organic matter, readily yielding to tillage and forming a fine seed bed. It was prepared by deep plowing, harrowing, and reducing the surface to fine tilth. The roots produced by a previous year's growth when not in cultivation were carefully removed from the soil. The physical condition of the soil at the time of planting was all that could be desired, and the growth of the beets was uniform and luxuriant. The beets were planted on May 5, and thinned to nearly the proper stand about June 15, the thinning being completed about one month later. They were cultivated once a week until July 15. The meteorological data for Washington for the period of growth is as follows:

Meteorological data for Washington, D. C., from May to October, 1900.

Month.	Mean tempera- ture.	Precipi- tation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May	64.4	4.02	258.0	443.8	58	17	9
June	72.2	10.94	244.1	445.9	55	11	9
July	78.7	1.25	351.6	453.0	78	18	5
Total	71.8	16.21			63.7	46	23
August	79.6	2.28	334.7	423.2	79	22	5
September	73.6	4.61	254.6	373.4	68	15	6
October	61.6	1.44	168.7	346.0	49	13	14
Total	71.6	8.33			65.3	50	25
Sum total	71.7	24.54			64.50	96	48

The dates of securing samples, the weight of samples obtained, the estimated yield in tons per acre, average weight of the beets har-

vested, the percentage of sugar in the beet, and the purity of the juice are given in the following table:

Analytical and field data on the Austrian Special Kleinwanzlebener beets grown on the experiment farm, Potomac Flats.

Date harvested.	Weight harvested from 5 feet of row.	Estimated tons per acre.	Average weight.	Sugar in the beet.	Purity.
1900.	<i>Lbs. Oz.</i>		<i>Ounces.</i>	<i>Per cent.</i>	
September 19	46 8	13.5	14.6	9.8	
September 24	49 2	14.3	17.9	9.3	72.1
October 3	52 5	15.2	16.1	8.7	70.8
October 9	56 7	16.4	15.8	8.4	71.5
October 15	50 14	14.8	17.7	8.4	71.0
October 22	61 3	17.8	19.1	7.9	69.2
October 30	60 2	17.5	19.8	7.6	68.4
November 6	58 8	17.0	17.7	7.9	63.4
November 13	40 11	11.8	13.6	7.8	69.0
November 19	46 6	13.5	17.7	8.6	71.1
November 27	44 7	12.9	20.3	8.1	66.9
December 3			27.8	7.1	66.4
December 11			22.5	8.6	69.0
Average		15.0	18.5	8.3	69.1

As seen by the table, the samples were taken at intervals of from five to seven days, beginning on September 19 and ending on December 11. The samples which were taken on December 3 and 11 were not weighed, nor was there any calculation made of the yield per acre. The average weight of the beets harvested, however, was determined as usual in these two samples. The average data show that the beets weighed 18.5 ounces, had a content of 8.3 per cent of sugar, with a purity of 69.1. The highest content of sugar was found in the beets first harvested, which leads me to believe that had the analyses been commenced at an earlier date, as, for instance, the first of September or the last week in August, a higher content of sugar might have been found. During the latter part of October there was a notable loss in sugar contained in the samples, which, however, was regained in November and continued until the advent of heavy frosts, a little before the middle of December. The variation in the content of sugar in the individual samples from the mean is quite small; the content of sugar having remained almost constant during the whole period of the investigation. The purity coefficients are very low, but probably no lower than would be expected in beets having only a little over 8 per cent of sugar and produced in a soil favorable to rapid and spongy growth. The analytical data show the futility of attempting to grow beets for commercial purposes in such an environment as was afforded by the Department farm.

EXPERIMENTS CONDUCTED BY THE INDIANA STATION.

These experiments were made at two stations, namely, at the agricultural experiment station at Lafayette and at North Judson, in Starke County. Mr. Huston, the chemist of the station, in his report dated November 7, 1900, makes the following statements:

The beets on our own farm, Lafayette, were badly affected by leaf spot. Those at North Judson were much better, but in sugar percentage they did not equal the beets that we have been receiving from North Judson, which were raised from commercial seed. I have asked Mr. Wilson to send me additional samples, which ought to be here in a few days, and we shall continue examining beets on our own farm until the ground freezes. The season has been unusually warm and pleasant; in fact I think a little too warm in this section for beets to ripen. We had no killing frost until after the 1st of November, and we have not yet had a frost hard enough to injure green tomatoes. The ground is fairly moist, so that the beets are probably still growing. This is a very unusual state of circumstances, as our first killing frost is due, on an average, by the 15th of September.

The meteorological data for Lafayette from May to October are as follows:

Meteorological data for Lafayette,¹ Ind., from May to October, 1900.

	Mean tempera- ture.	Precipi- tation.	Clear days.	Cloudy days.
	Degrees.	Inches.		
May.....	64.3	6.89	8	14
June.....	69.8	7.88	2	17
July.....	74.5	5.36	14	3
Total.....	69.5	20.13	24	34
August.....	78.6	4.21	13	7
September.....	69.3	2.75	13	5
October.....	62.2	3.43	14	7
Total.....	70.0	10.39	40	19
Sum total.....	69.75	30.52	64	53

¹For sunshine data see table for Indianapolis, the nearest station at which sunshine records were kept.

Meteorological data for Indianapolis,¹ Ind., from May to October, 1900.

	Mean tempera- ture.	Precipi- tation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	Degrees.	Inches.	Hours.	Hours.			
May.....	65.6	6.14	289.7	446.7	65	7	11
June.....	71.0	4.42	241.7	449.0	54	6	9
July.....	75.2	4.10	325.3	455.2	71	14	4
Total.....	70.6	14.66			63.3	27	24
August.....	79.4	3.32	309.5	425.2	73	9	4
September.....	70.8	2.95	220.8	373.6	59	10	7
October.....	63.4	3.20	226.1	344.9	66	15	7
Total.....	71.2	9.47			66.0	34	18
Sum total.....	70.9	24.13			64.65	61	42

¹ Fifty-nine miles southeast of Lafayette.

One analysis was made of the beets grown at the experiment station, with the following results:

Date of planting.....	May 7
Date of harvesting.....	October 29
Average weight.....	ounces.. 4.9
Sugar in juice.....	per cent.. 10.5
Sugar in beet.....	do.... 9.9
Purity.....	83.0

Two analyses were made of the beets grown by J. M. Wilson at North Judson, giving the following results:

Field data and analyses for Austrian Special Kleinwanzlebener grown at North Judson, Ind.

No.	Date of planting.	Date of harvest.	Average weight.	Sugar in juice.	Sugar in beet.	Purity.
			<i>Ounces.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
1	May 26.....	Oct. 1.....	17.5	13.7	13.0	86.0
2	May 26.....	Oct. 24.....	12.9	15.1	14.3	93.0

The only meteorological data obtainable for North Judson are those for South Bend, as given below, and as no regular station, either agricultural or meteorological, exists at these points, the results for North Judson have not been included in the data platted on the charts.

Meteorological data for North Judson,¹ Ind., from May to October, 1900.

Month.	Mean temperature.	Precipitation.
	<i>Degrees.</i>	<i>Inches.</i>
May.....	62.9	1.74
June.....	69.0	2.66
July.....	72.6	5.81
Total.....	68.2	10.21
August.....	76.4	6.43
September.....	67.4	2.25
October.....	61.4	1.31
Total.....	68.4	9.99
Sum total.....	68.3	20.20

¹ Data given is for South Bend, 40 miles northeast of North Judson. For sunshine record see Lafayette, 58 miles south, and Indianapolis, about 100 miles south, of North Judson.

In transmitting the above meteorological data Mr. Huston, under date of March 27, 1901, makes the following comments:

I inclose the weather record for Lafayette and for South Bend. We have no observer at North Judson, but South Bend is up the valley a little ways, and is practically on the same isotherm. You will notice that the season was quite abnormal at both places, and especially is this true of part of the season during which time the beets ought to ripen; and the number of cloudy days is unusually high. While the rainfall during September and October in the northern part of the State is somewhat below normal, the excessively high temperature in October, together with the fact that nearly all of the rainfall occurred on October 6 and 7, which furnished plenty of water to keep the beets growing, made the ripening period even less favorable than the record would seem to indicate.

It will be observed that the beets grown at North Judson, although somewhat small, were of fair sugar content and of high purity. The beets grown at the experiment station farm were phenomenally small and contained a low percentage of sugar, but a purity slightly above the minimum standard desirable for manufacturing purposes. It is remarkable to see so great a difference in the composition of beets grown in the same State and in localities less than 100 miles apart. North Judson is almost exactly north of Lafayette, and its proximity to Lake Michigan doubtless accounts for the differences in the meteorological environment of the two places. The experience of former

years in the same localities shows that Starke County, in which North Judson is situated, is favorably located for the production of beets of high grade.

EXPERIMENTS CONDUCTED BY THE IOWA STATION.

The sample of Austrian Special Kleinwanzlebener was planted on May 29 in rows 16 inches apart, and thinned on June 20. The plot received the usual careful cultivation. The assistant in agriculture at the Iowa station at Ames, Mr. Atkinson, gives the following description of the soil on which the experiments were made:

The soil upon which the Austrian Special Kleinwanzlebener were grown was an upland prairie. It was a clover sod in 1898; in 1899 it grew a crop of spring wheat, while the beets were grown on it in 1900. It has been several years since it received an application of manure.

The samples were harvested on November 6, and eight beets were sent to the Department of Agriculture, and were analyzed on November 12 with the following results:

Average weight	ounces...	13
Sugar in the beet	per cent...	11.7
Purity		76.9

The season was not considered a favorable one for beet culture. The climatic conditions prevailing during the growing season are shown in the accompanying tables:

Meteorological data for Ames, Iowa,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
	<i>Degrees.</i>	<i>Inches.</i>		
May	64.4	4.36	14	5
June	69.4	6.48	19	1
July	73.0	9.14	12	0
Total	68.9	19.98	45	6
August	76.8	5.46	16	0
September	66.0	7.12	11	1
October	59.8	3.73	17	4
Total	67.5	16.31	44	5
Sum total	68.2	36.29	89	11

¹ For sunshine record see table for Des Moines, that being the nearest station at which a sunshine record was kept.

Meteorological data for Des Moines, Iowa,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Percent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May	64.2	4.76	274.6	451.9	61.0	9	6
June	70.1	4.89	339.3	456.2	74.0	12	2
July	74.2	5.15	300.1	461.8	65.0	11	5
Total	69.5	14.80			66.7	32	13
August	77.7	8.02	278.8	429.4	65.0	11	4
September	65.4	3.66	203.1	374.5	54.0	10	12
October	60.4	3.08	195.7	342.5	57.0	10	9
Total	67.8	14.76			58.7	31	25
Sum total	68.65	29.56			62.7	63	38

¹Thirty miles south of Ames.

EXPERIMENTS CONDUCTED BY THE KENTUCKY STATION.

The Austrian Special Kleinwanzlebener was planted April 25 in rows 18 inches apart, and was thinned May 22. The soil was a rich loam, and the estimated yield per acre was 10 tons. The season was reported as being favorable, the climatic conditions for the period of growth having been as follows:

Meteorological data for Lexington, Ky., from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May	65.6	3.54	297.6	441.7	67.00	15	7
June	72.5	2.19	291.6	443.1	66.00	4	7
July	77.0	2.80	358.4	450.1	80.00	15	3
Total	71.7	8.53			71.00	34	17
August	79.3	5.75	361.8	422.1	87.00	8	2
September	73.2	1.85	292.1	373.0	78.00	11	4
October	64.9	.79	243.5	347.3	70.00	15	5
Total	72.5	8.39			78.30	34	11
Sum total	72.1	16.92			74.65	68	28

The analyses were commenced by the station on August 30, and continued at intervals until October 16. After that time the analyses were suspended until November 19, when an additional sample was examined, nine series of analyses having been made altogether.

The results of these analyses were as follows:

Station analyses of Austrian Special Kleinwanzlebener grown at Lexington, Ky.

Date of sampling.	Number of beets taken.	Weight topped.	Weight trimmed.	Brix.	Sugar by polarization.	Sugar in beet.	Purity.
		<i>Ounces.</i>	<i>Ounces.</i>	<i>Degrees.</i>	<i>Per cent.</i>	<i>Per cent.</i>	
1900.							
August 30	2	11½	8	12.7	10.05	9.6	79.20
September 19	1	20½	15½	11.2	8.40	8.0	74.10
Do.	1	13½	10	10.0	7.00	6.6	70.00
September 26	2	8½	5½	10.2	6.85	6.6	67.72
October 6	1	14½	11	13.3	9.90	9.4	74.40
Do.	1	9½	6½	11.9	8.50	8.1	72.20
October 12	1	12½	7½	10.7	7.35	7.0	68.70
October 16	1	10½	8½	11.9	8.05	7.7	67.60
November 19		10½	8½	13.7	7.00	6.6	65.20
Average		12.47	9	11.73	8.12	7.7	71.01

A sample of these beets was sent to the Department of Agriculture for analysis on November 19. In forwarding this sample Dr. Peter, chemist of the station, calls attention to the fact that his analyses show that the beets deteriorated greatly after August. The data obtained by the analysis of the sample sent to the Department of Agriculture were as follows:

Average weight of the beets	ounces..	9
Percentage of sugar in the beets	per cent..	7.9
Purity		68.0

The data show that in the analysis of these samples at the Department of Agriculture a somewhat higher percentage of sugar, of a slightly increased purity, was obtained than that given for the samples taken on the same date, November 19, and analyzed at the Kentucky station. A part of the increase in sugar may be ascribed to the drying out of the samples in transit.

EXPERIMENTS CONDUCTED BY THE MICHIGAN STATION.

A field of sandy loam, selected for this experiment, was subsoiled about ten days before sowing the beet seed, and the surface of the field reduced to a proper degree of tilth. The seeding was done on April 28, 1900, and the beets received the usual cultivation to keep the surface of the soil loose and free from weeds.

The agriculturist of the station, Mr. B. D. Towar, reports:

We are very well pleased with the results, as the ground was by no means the most desirable for growing beets. A good season has been favorable to Michigan beets and satisfactory reports are coming from all directions.

The meteorological conditions are shown by the following data:

Meteorological data for Agricultural College, Mich.,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
	<i>Degrees.</i>	<i>Inches.</i>		
May.....	58.8	4.17	10	9
June.....	65.2	2.57	16	6
July.....	69.6	4.15	13	6
Total.....	64.5	10.89	39	21
August.....	73.3	2.98	12	3
September.....	63.2	.89	16	8
October.....	56.6	2.77	14	8
Total.....	64.4	6.64	42	19
Sum total.....	64.45	17.53	81	40

¹ For sunshine record see table for Detroit, the nearest station at which this record is kept.

Meteorological data for Detroit, Mich.,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Percent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May.....	60.0	3.08	246.5	451.9	55	6	13
June.....	66.6	3.99	310.6	456.2	68	12	6
July.....	72.0	3.71	267.6	461.8	58	9	7
Total.....	66.2	10.78			60.3	27	26
August.....	75.5	2.08	263.3	429.4	61	12	7
September.....	66.8	1.88	215.2	374.5	57	10	5
October.....	60.0	2.85	191.0	342.5	56	12	6
Total.....	67.4	6.81			58	34	18
Sum total.....	66.8	17.59			59.15	61	44

¹ Seventy-six miles southeast of Agricultural College.

Analytical data obtained with the Austrian Special Kleinwanzlebener at the Michigan station.

Number of sample.	Seed test from 100 seed bulbs.			Average distance apart in row.	Yield per acre.	Average weight samples analyzed.	Per cent of sugar in juice.	Per cent of sugar in beet.	Purity.
	Number of sprouts at the end of one week.	Number of sprouts at the end of two weeks.	Seed balls that did not grow.						
1.....	122	150	30	<i>Inches.</i> 6+	<i>Tons.</i> 15.21	<i>Pounds.</i> 12	13.61	12.9	79.27
2.....	155	175	25	6—	16.29	12	14.03	13.3	80.77
Total ..					31.50	24	27.64	26.2	160.04
Average ..					15.75	12	13.82	13.1	80.02

EXPERIMENTS CONDUCTED BY THE NEW YORK STATION AT GENEVA.

The report of the collaborative work done at the Geneva station was made by the agriculturist, Mr. G. W. Churchill, under date of December 7, 1900. Twelve different plots of beets were grown, of which numbers 3, 6, 9, 12, and 13 were of the Austrian Special Kleinwanzlebener. Plot No. 13 was composed of check rows planted between plots which were fertilized with different materials in order that there should be no extension of the effect of the fertilizers from one plot to another. Under date of December 19 Director Jordan, of the station, made the following report:

For three years we have been making an effort to compare the effect of commercial fertilizers with that of farm manures upon the composition of sugar beets. In all of these years the percentage of sugar and the coefficient of purity with the beets raised on the farm manure have been of a high standard, the percentage of sugar in two years being higher than where the beets received commercial fertilizers. The other year the percentage of sugar was higher in the beets fertilized with farm manure than where no farm manure at all was used. The amount of manure per acre was 40,000 pounds, or about 10 cords. It was manure from the cow stable which had been somewhat fermented but not very fully. In other years we have used fresh manure. I am now working up the results for the three years to publish in a bulletin. I am inclined to think that we have all the time been placing altogether too much confidence in German results as applied to American conditions.

The following table gives the mean temperatures and precipitations for Lyons, 13 miles north of Geneva, during the growing season, the sunshine data not being obtainable for this or any other station nearer than Ithaca, for which place a full set of data may be found further on.

Meteorological data for Lyons, N. Y., from May to October, 1900.

Month.	Mean temperature.	Precipitation.
	Degrees.	Inches.
May	58.4	1.85
June	67.9	2.74
July	72.4	3.46
Total.....	66.2	8.05
August	73.8	2.49
September	67.1	2.01
October	58.6	3.71
Total.....	66.5	8.21
Sum total	66.4	16.26

The samples sent to us by the Geneva station were analyzed in the Division of Chemistry, and the results obtained with the Austrian Special Kleinwanzlebener were as follows:

Department analysis of Austrian Special Kleinwanzlebener beets grown at Geneva, N. Y.

Number.	Weight.	Sugar in juice.	Sugar in beet.	Purity.
	Pounds.	Per cent.	Per cent.	
1	17	15.2	14.4	82.2
2	17	16.0	15.2	83.8
3	17	16.4	15.6	82.8
4	16	16.1	15.3	84.3
5	17	15.2	14.4	82.2
Average.....	16.8	15.78	14.98	83.06

In the above data the samples represented by Nos. 1 and 2 were grown under a heavy fertilization of superphosphate. Nos. 3 and 4, corresponding to Nos. 9 and 12 referred to above, were grown under fertilization with farmyard manure, and No. 5, corresponding to No. 13, represents the check rows planted between the fertilized plots.

A complete study of the sugar content of the beets grown at the Geneva station was made at that place between the dates of November 23 and December 6. The results of these determinations are given in the following table:

Austrian Special Kleinwanzlebener beets.

[Grown and analyzed at the New York Experiment Station, Geneva, N. Y.]

Date of analysis.	Number of plat.	Number of beets used for analysis.	Degree Brix.	Average weight of beets (without tops).	Sugar in juice.	Sugar in beet.	Coefficient of purity.
				Pounds.	Per cent.	Per cent.	
1900.							
November 23	8	20	19.8	0.97	16.8	16.0	84.8
November 24	8	20	20.7	.965	17.6	16.7	85.2
November 23	9	20	18.8	.95	15.7	14.9	83.7
November 24	9	20	18.6	1.00	15.7	14.9	84.3
November 23	10	20	19.4	.82	16.6	15.8	85.4
December 1	10	20	19.7	.89	17.1	16.3	87.0
November 23 ¹	11	20	22.1	.79	18.5	17.6	84.0
December 4	11	20	20.9	.91	17.5	16.6	83.9
November 23	12	20	19.0	1.63	16.1	15.3	84.7
December 11	12	20	18.9	.89	16.1	15.3	85.3
December 4	Check.	20	20.9	.76	17.5	16.6	83.6
December 6	Check.	12	19.0	.88	16.2	15.4	85.1
Average			19.8	2.95	16.8	16.0	84.8

¹ Beets somewhat wilted.

² Equivalent to 15.2 ounces.

EXPERIMENTS CONDUCTED BY THE NEW YORK STATION AT ITHACA.

Director I. P. Roberts, of the Ithaca station, reported under date of March 11, 1901, that the Austrian Special Kleinwanzlebener beet seed for the collaborative experiments had been planted on May 17, 1900, in rows 20 inches apart, and harvested on October 26. The soil was a sandy loam in a good state of fertility. The record of the climatic conditions during the growing season is as follows:

Meteorological data for Ithaca, N. Y., for May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May	57.1	1.51	281.9	451.9	62	11	15
June	67.0	1.98	242.1	456.2	75	7	12
July	72.0	2.41	332.1	461.8	72	3	11
Total	65.4	5.90	314.8	429.4	69.7	21	38
August	72.3	2.93	260.4	374.5	73	9	8
September	64.9	.94	216.0	342.5	70	11	9
October	57.2	4.06			63	9	13
Total	64.8	7.93			68.7	29	30
Sum total.....	65.1	13.83			69.2	50	68

The analytical data obtained at the station on the beets raised were as follows:

Average weight of beets analyzed	ounces..	18
Yield per acre.....	tons..	15
Sugar in the juice.....	per cent..	14.8
Sugar in the beet.....	do...	14.0
Coefficient of purity		81.9

EXPERIMENTS CONDUCTED BY THE NORTH CAROLINA STATION.

The seed of the Austrian Special Kleinwanzlebener were planted on May 22, the date of thinning was June 15, and the date of harvesting November 1. The soil was a sandy loam, and the width between the rows 36 inches. The season was rather dry and to that extent unfavorable. The estimated yield per acre was $1\frac{1}{4}$ tons.

Samples of the beets were forwarded to this Department from Raleigh on November 21. They were, however, in such a poor condition when received that they were deemed worthless for analytical purposes and the analytical data are confined therefore to the analyses made at the station at Raleigh. The figures obtained are as follows:

Average weight of beets	ounces..	12.4
Average sugar in the beets	per cent..	5.2

The above data show that the beets were remarkably poor even for the locality. While it was not expected that they would show a

high quantity of sucrose, it is evident that under other conditions or in other seasons a much better result could be obtained. The following table shows the meteorological conditions under which these beets were grown:

Meteorological data for Raleigh, N. C., from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May	69	3.12	335.1	436.1	77	16	3
June	76	8.47	284.9	437.2	65	9	7
July	80.9	5.53	313.5	444.3	71	16	4
Total	75.3	17.12			71	41	14
August	82.2	4.51	357.4	418.7	85	25	1
September	75.8	2.13	316.5	372.2	85	20	1
October	65.6	1.04	208	348.9	60	14	8
Total	74.5	7.68			76.7	59	10
Sum total	74.9	24.80			73.9	100	24

EXPERIMENTS CONDUCTED BY THE UTAH STATION.

The data from the Utah Agricultural Experiment Station are included in this report not so much for the purpose of direct comparison as to show the influence of irrigation upon the yield and character of the beets. It is evident that the artificial conditions which obtain in irrigated regions are so different from those that naturally exist as to render of doubtful value a comparison between the two sets of data, and hence the results in Utah have not been included in the graphic charts. It is proposed at some future time to make an additional study in irrigated regions of the influences of sunshine and the differences of latitude on the character of the beets grown under irrigation. Nevertheless there will be some interest attached to the utilization of the data from Utah in the present comparison.

The beets were planted on May 8, the date of thinning was June 7, the dates of irrigation, June 22, July 14 and 28, August 11, and September 1; the dates of cultivation were June 20 and 25, July 16, August 2 and 14; the dates of harvesting were October 24, 25, and 27.

The yield per acre and the analytical results as determined at the Logan station are as follows:

Estimated yield per acre.....	tons..	18.9
Percentage of sugar in the beet	per cent..	12.1
Purity coefficient.....		84.2

The climatic conditions under which these beets were grown are shown in the following tables of data for Logan and Salt Lake City, Utah:

Meteorological data for Logan, Utah,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Clear days. ²	Cloudy days. ²
	<i>Degrees.</i>	<i>Inches.</i>		
May.....	57.4	1.42	17	11
June.....	70.4	.19	25	3
July.....	70.9	.51	26	3
Total.....	66.2	2.12	68	17
August.....	69.4	.71	24	3
September.....	59.9	.94	20	6
October.....	49.8	2.38	26	2
Total.....	59.7	4.03	70	11
Sum total.....	62.95	6.15	138	28

¹ For sunshine data see table for Salt Lake City, the nearest point at which this record was kept.

² These figures are for Corinne, 19 miles southwest of Logan.

Meteorological data for Salt Lake City, Utah,¹ from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Sunshine.			Clear days.	Cloudy days.
			Actual.	Possible.	Per cent.		
	<i>Degrees.</i>	<i>Inches.</i>	<i>Hours.</i>	<i>Hours.</i>			
May.....	60.6	0.44	356.5	449.1	79	21	3
June.....	73.8	.08	414.1	451.9	92	28	0
July.....	74.7	.32	430.5	458.6	94	26	0
Total.....	69.7	.84			88.3	78	3
August.....	74	.72	357.6	427.4	84	23	0
September.....	62.7	1.44	288.8	374.	77	18	1
October.....	52.6	1.99	208.8	343.9	61	17	10
Total.....	63.1	4.15			74	58	11
Sum total.....	66.4	4.99			81.2	136	14

¹ Sixty-six miles south of Logan.

DISCUSSION OF IRRIGATION EXPERIMENTS.

The director of the station, Mr. John Widtsoe, under date of November 17, makes the following comments on the season's work:

The sugar content * * * is much lower than the average of the beets grown at the station in the previous years. The beets grown in the irrigation experiments have this season contained from 15 to 18 per cent of sugar. The above varieties were treated as nearly as possible the same as the average beet grower of Utah treats his crop. It is difficult to account for the low per cent of sugar, though I am led to believe from the experiments carried on at the station that the amount of water used and the time of its application has a great influence on the composition of the beet.

It is evident from the various experiments in irrigation previously conducted that an elaborate series of comparative studies should be made to determine, first, the amount of water necessary to produce the maximum weight of crop and content of sugar, and second, the

most favorable periods of its application. It is doubtful whether it is advisable to apply an abundant quantity of water to a beet field as late as September 1, as was the case in the above experiments. The tendency in this case would be to keep the beets growing too long, or perhaps to start a second growth, which would consume much of the sugar already stored.

Another point in connection with the irrigation work should be prominently kept in mind, viz, that the injurious effects of high temperatures as shown in the inferior content of sugar in beets grown far south of the natural habitat may be due rather to premature ripening and the rapid evaporation of water than to the effect of temperature directly. It is difficult to understand how the rise of a few degrees of temperature could so seriously affect the sugar content of the beet simply by reason of increased amount of heat. To me it seems more probable that the deleterious influences of a high temperature are of an indirect rather than a direct nature.

EXPERIMENTS CONDUCTED BY THE WISCONSIN STATION.

CULTURAL DATA.

The field notes in regard to the cultivation of the Austrian Special Kleinwanzlebener beet seed, furnished by Mr. Roscoe H. Shaw, acting chemist of the Madison station, under date of November 7, 1900, are as follows:

The field set apart for sugar-beet work at the Wisconsin experiment station this season was a plat of clay loam consisting of half an acre. This plat had been used for the sugar-beet work in 1898 and for variety tests in 1899. The field was not manured on the northern half in 1898, and not at all in 1899.

The plat was divided into quarters, the middle half receiving a mixed fertilizer consisting of 90 pounds of Star phosphate (dissolved bone), 90 pounds of sulphate of potassium, and 100 pounds of nitrate of soda. The phosphate was put on first and the potash and half of the nitrate mixed and distributed next. The northern quarter and southern quarter were left unfertilized. The field was disked north and south and then dragged with pulverizer east and west. Eight varieties of beet seed were planted, all of which were received from the Department of Agriculture. The field was planted on May 15. A heavy shower, followed by six days of dry weather, caused the plat to bake so hard that with the exception of the section devoted to Zehringen (Strandes, Germany), of which there was no seed left, it was reharrowed and replanted. From this circumstance, owing to lack of seed, but seven rows of the Austrian Special Kleinwanzlebener were planted; these rows were made 15 inches apart. The second planting was finished May 21.

May 26.—The section not replanted was cultivated with garden wheel hoe. At this time the crust was very hard and beet plants were coming up in spots.

May 31.—No rain to this date and surface of ground was dried out, but beet sprout up in rows.

June 1.—A good rain fell for about an hour.

June 2.—Whole plat cultivated with wheel hoe.

June 4.—Beet plants well up; 1 to 2 inches high.

June 11.—The work of thinning beets was begun. They were thinned so as to

have one strong plant every 8 or 9 inches in the row. At this date the Austrian Special Kleinwanzlebener was rather behind the other varieties in development.

June 19.—The remaining 50 pounds of nitrate, mixed with twice the amount or more of dirt from the field, was sown on the fertilized part.

June 24.—No difference was noticeable between different varieties or between the fertilized or unfertilized parts.

August 4.—Fertilized half of field seemed to be in better condition than the unfertilized.

August 18.—No apparent difference was noticeable between varieties.

October 13.—The beets were harvested; they were rather smaller than average beets and all of the varieties were of comparatively uniform size.

The section of the field devoted exclusively to the Austrian Special Kleinwanzlebener was 192 feet long by 8.75 feet wide, containing 1,680 square feet.

In harvesting beets 50-pound samples from each of the three sections of each variety were taken. In these samples the percentage of shrinkage was determined, and a subsample selected for the sugar determination.

Austrian Special Kleinwanzlebener beets grown and analyzed at the Wisconsin Experiment Station, Madison, Wis.

	Fertilized.	Unfertilized.
Average weight.....pounds..	0.81	0.73
Shrinkage ¹per cent..	18.98	19.07
Specific gravity.....	1.0779	1.0753
Sugar in the juice.....per cent..	16.66	15.28
Purity.....	88.53	83.93
Sugar in the beet.....per cent..	15.81	14.52
Total yield:		
Total weight.....pounds..	841.6	542.9
Weight less shrinkage.....do....	681.9	439.4
Sugar.....do....	107.6	63.81

¹ The shrinkage refers to that part of the beet lost in washing and crowning.

Results calculated to the acre are as follows:

	Fertilized.	Unfertilized.
	<i>Tons.</i>	<i>Tons.</i>
Total weight.....	10.91	7.04
Weight less shrinkage.....	8.84 ¹	5.7
Sugar.....	1.398	0.83

It is interesting to note the influence of the fertilizer employed. The fertilized sections of the plat yielded almost 4 tons more per acre than the unfertilized, while the percentage of sugar in the fertilized portion was considerably higher than in the unfertilized. Since it would not be quite fair to select either the fertilized or the unfertilized data for purposes of comparison, it has been determined to take the mean of the two as representing the proper data for the comparative work.

The meteorological conditions under which these beets were grown are shown by the following table:

Meteorological data for Madison, Wis., from May to October, 1900.

Month.	Mean temperature.	Precipitation.	Clear days.	Cloudy days.
	<i>Degrees.</i>	<i>Inches.</i>		
May.....	60.6	1.86	6	14
June.....	67.7	3.20	9	7
July.....	70.2	6.91	8	14
Total.....	66.2	11.97	23	35
August.....	75.2	2.72	9	10
September.....	63.6	2.89	6	12
October.....	58.0	4.43	10	14
Total.....	65.6	10.04	25	26
Sum total.....	65.9	22.01	48	71

SUMMARY.

Summary of averages of analytical data, 1900.

	Weight.	Yield per acre.	Sugar in the beet.	Coefficient of purity.
	<i>Ounces.</i>	<i>Tons.</i>	<i>Per cent.</i>	
Raleigh, N. C.....	12.4	1.3	5.2	
Lexington, Ky. ¹	9.0	10.0	7.8	69.5
Washington, D. C.....	18.5	15.0	8.3	69.1
Lafayette, Ind.....	4.9		9.9	83.0
Ames, Iowa.....	13.0		11.7	76.9
Logan, Utah.....		18.9	12.1	81.2
Agricultural College, Mich.....	12.0	15.8	13.1	80.0
North Judson, Ind.....	15.2		13.7	89.5
Ithaca, N. Y.....	18.0	15.0	14.0	81.9
Madison, Wis. ²	12.3	9.0	15.2	86.2
Geneva, N. Y. ³	16.1		15.5	83.9

¹ Average of data obtained at Washington and at Lexington.

² Average of data for fertilized and unfertilized plats.

³ Average of data obtained at Washington and at Geneva.

Summary of meteorological data, May to October, 1900.

	Temperature.	Precipitation.	Clear days.	Cloudy days.	Sunshine.
	<i>Degrees.</i>	<i>Inches.</i>			<i>Per cent.</i>
Raleigh, N. C.....	74.9	24.8	100	24	73.9
Lexington, Ky.....	72.1	16.9	68	28	74.7
Washington, D. C.....	71.7	24.5	96	48	64.5
Lafayette, Ind. ¹	69.8	30.5	64	53	64.7
Ames, Iowa ²	68.2	36.3	89	11	62.7
Logan, Utah ³	63.0	6.2	138	28	81.2
Agricultural College, Mich. ⁴	64.5	17.5	81	40	59.2
North Judson, Ind.....	68.3	20.2	(⁵)	(⁵)	(⁵)
Ithaca, N. Y.....	65.1	13.8	50	68	69.2
Madison, Wis.....	65.9	22.0	48	71	
Geneva, N. Y. ⁶	66.4	16.3	(⁷)	(⁷)	(⁷)
	66.0	15.0			

¹ Approximate data observed at Indianapolis.

² Approximate data observed at Des Moines.

³ Approximate data observed at Salt Lake City.

⁴ Approximate data observed at Detroit, Mich.

⁵ See Lafayette.

⁶ Data for Lyons, N. Y., used.

⁷ See Ithaca.

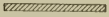
Summary of geodetic data for experiment stations.

	Average length of day.	Latitude.	Altitude.
	<i>H. M.</i>	<i>° ' "</i>	<i>Fect.</i>
Raleigh, N. C	14 7	35 48 00	363
Lexington, Ky.....	14 18	38 02 25	979
Washington, D. C.....	14 23	38 53 23	37.5
Lafayette, Ind.....	14 30	40 23 00	542
Ames, Iowa.....	14 38	42 02 00	917
Logan, Utah.....	14 37	41 44 00	4,506
Agricultural College, Mich ¹	14 42	42 45 00	847
North Judson, Ind.....	14 34	41 11 00	695
Ithaca, N. Y.....	14 41	42 27 00	810
Madison, Wis.....	14 44	43 04 36	955
Geneva, N. Y.....	14 44	42 53 00	453

¹ Determinations for Lansing, Mich.

CHART NO.1—SHOWING
PERCENTAGE OF SUGAR IN THE BEET,
LATITUDE OF STATION AND SUNSHINE RECORD.

EXPLANATION

PERCENTAGE OF SUGAR IN THE BEET —————
 PERCENTAGE OF SUNSHINE DURING PERIOD COVERED - - - - -
 NUMBER OF CLEAR DAYS IN THE MONTH    
 LATITUDE OF STATION - - - - -

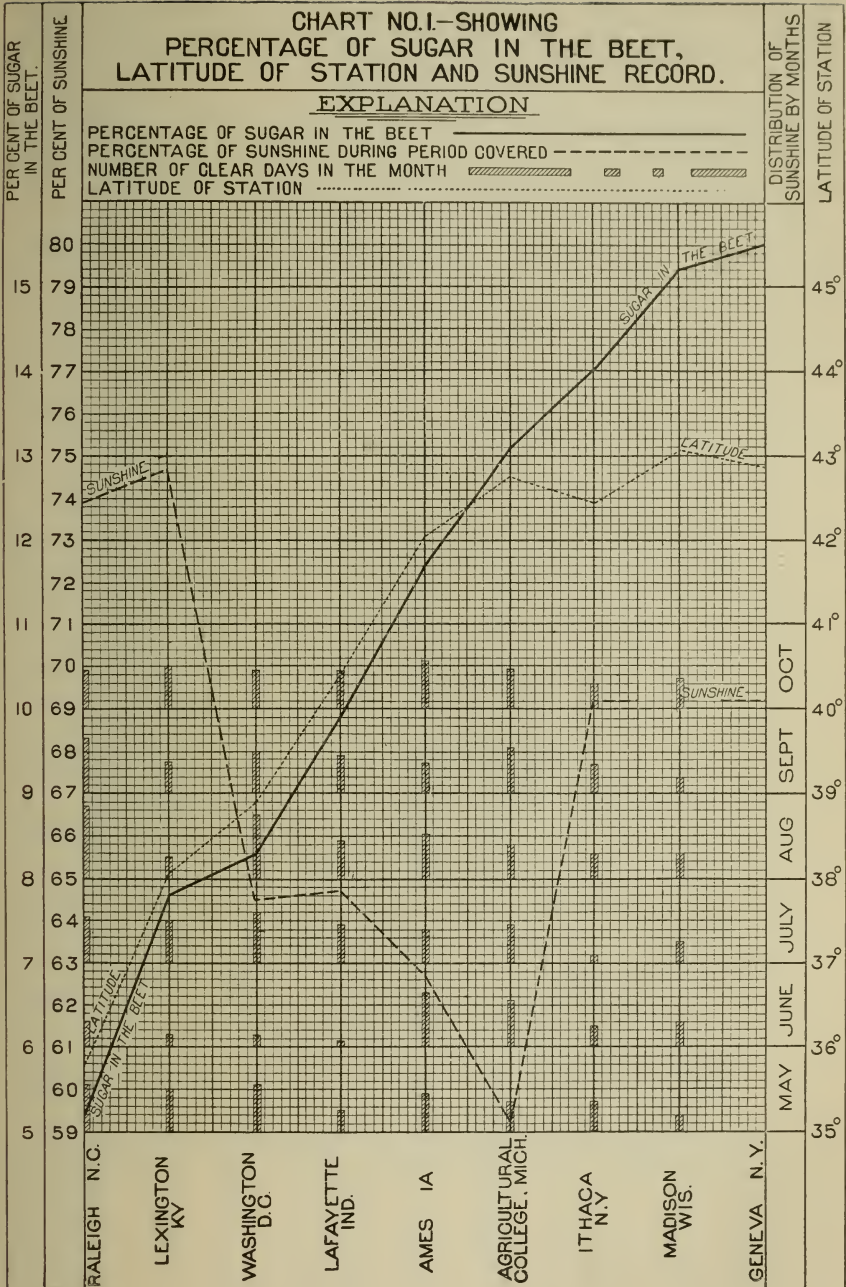


CHART NO.2—SHOWING PERCENTAGE OF SUGAR IN THE BEET, PURITY OF JUICE, TEMPERATURE AND AVERAGE LENGTH OF DAY AT STATION.

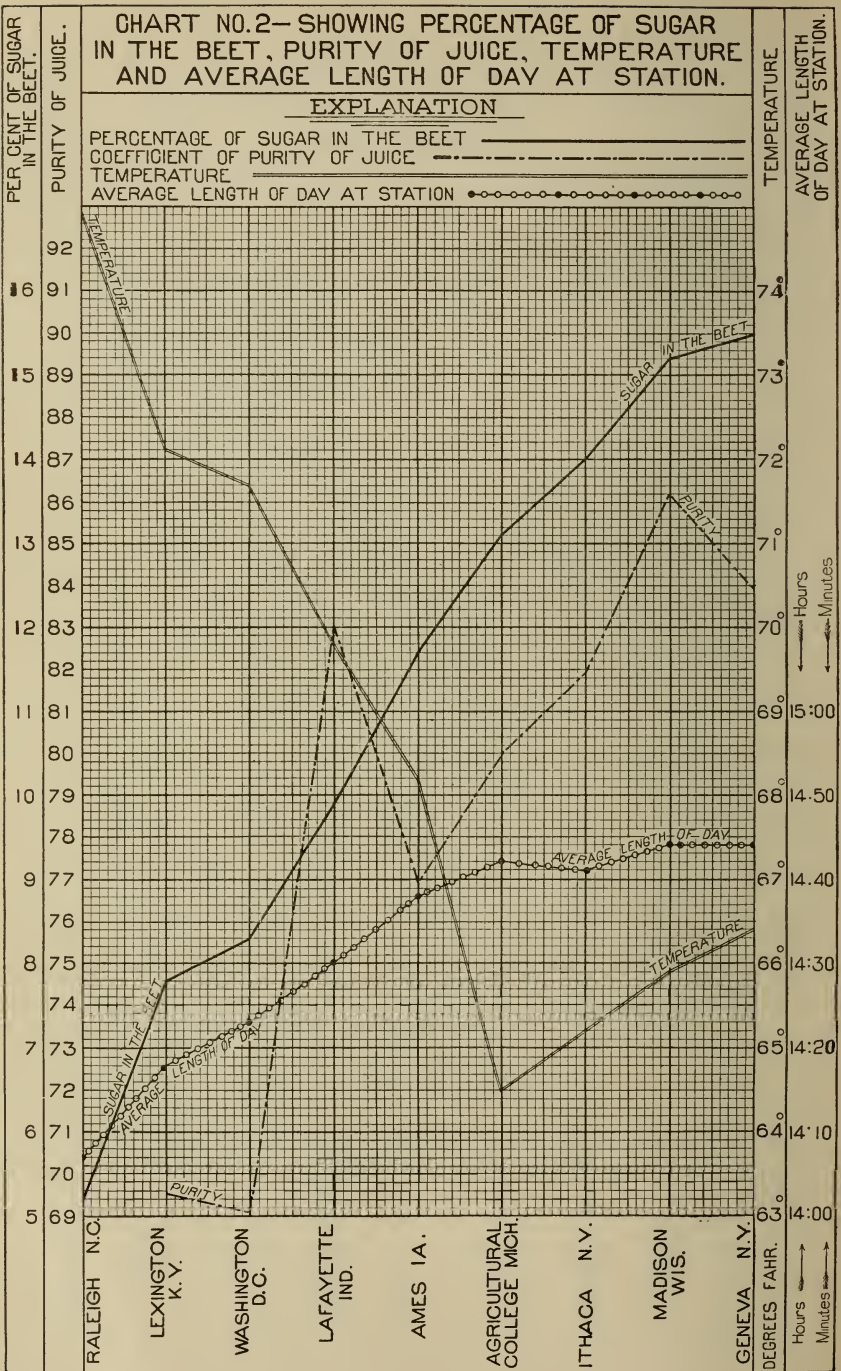
EXPLANATION

PERCENTAGE OF SUGAR IN THE BEET —————

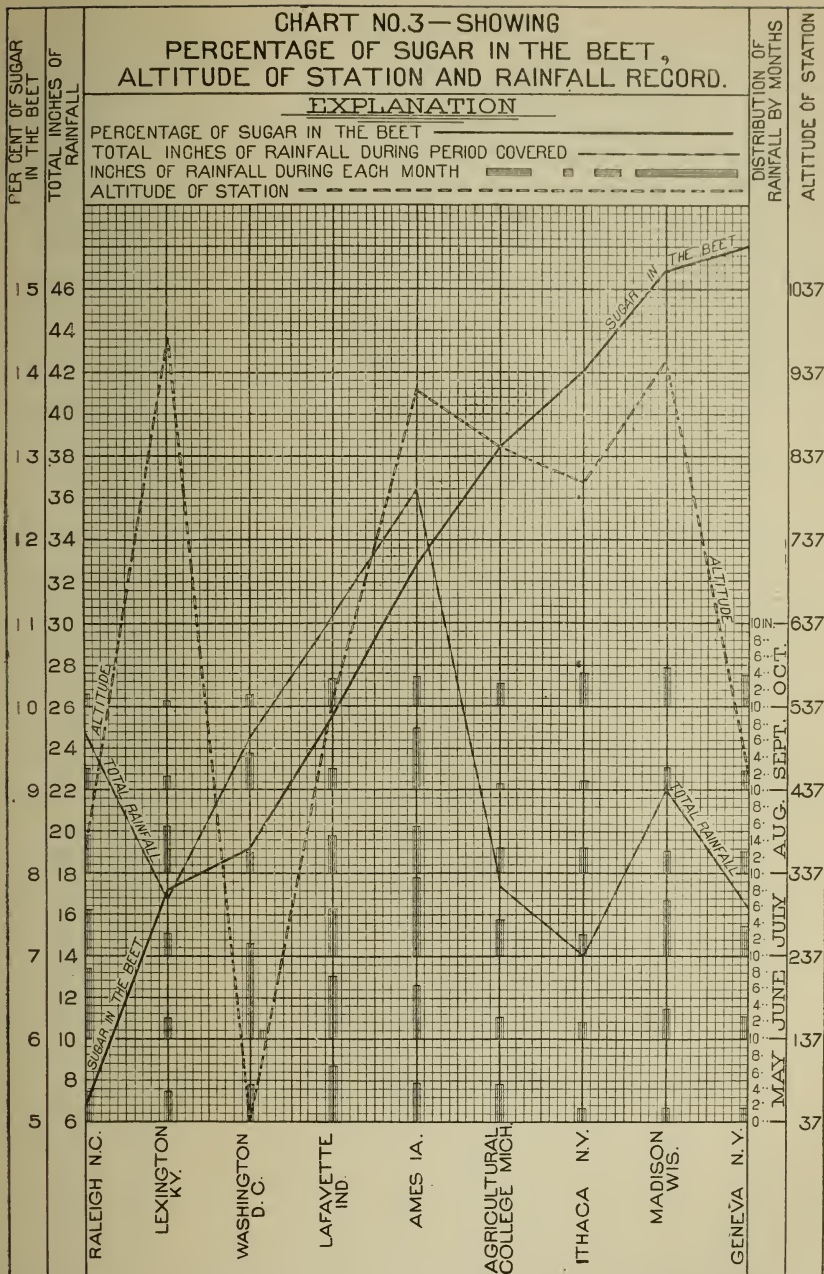
COEFFICIENT OF PURITY OF JUICE - - - - -

TEMPERATURE —————

AVERAGE LENGTH OF DAY AT STATION ○○○○○○○○○○○○○○○○○○○○○○○○○○○○○○○



PERCENTAGE OF SUGAR IN THE BEET _____
TOTAL INCHES OF RAINFALL DURING PERIOD COVERED _____
INCHES OF RAINFALL DURING EACH MONTH _____
ALTITUDE OF STATION _____



CONCLUSIONS.

The conclusions which are deduced from a study of the preceding data and the graphic charts, based as they are upon the observations of a single year, will be subject to such correction as may be indicated by the results of subsequent studies.

On Chart No. 1 are platted the percentage of sugar in the beet, the latitude of the station, and the hours of sunshine. The curve representing the latitude and the curve representing the sugar in the beet are evidently more nearly related to each other than the curves representing any other figures in any other one of the three charts. It will be seen that there is a very close agreement between the latitude curve and the percentage of sugar curve. High sugar and high latitude run very evenly together. The actual hours of sunshine do not appear to have much influence upon the sugar content, or perhaps it would be better to say that the curves do not coincide even approximately.

It is evident that the elements of sunlight, which are active in promoting the action of the chlorophyll cells in the formation of sugar, do not depend upon the direct rays of the sun. The diffused light coming through the clouds is apparently quite as effective as the direct light. The highest percentage of sunshine found in any of the stations plotted was at Lexington, Ky., reaching nearly 75 per cent of the possible hours of sunlight. The lowest percentage of direct sunshine was found at Agricultural College, Mich., being 59.2 per cent. Interesting data are also given in connection with the total sunshine by the boxed lines showing approximately the distribution of the sunshine in the various months, i. e., the number of clear days.¹ In order to show the complete relation, however, this line must be taken in connection with the number of cloudy and partly cloudy days. A striking illustration of this is shown by the data at Lexington, Ky., for June. During this month there were four clear days, and therefore the boxed line for June in the Lexington column is very short. The number of cloudy days was only seven, and there were nineteen days partly cloudy. In the study of the chart, therefore, it must be remembered in all cases that the boxed lines represent only the number of perfectly clear days. In Chart No. 1, therefore, it is seen that Raleigh, N. C., and Washington, D. C., have the largest number of clear days, while Madison, Wis., Ithaca, N. Y., Lafayette, Ind., and Lexington, Ky., are types of stations where the number of clear days was relatively small. No record was made for the station of Geneva, but the data may be assumed to be practically the same as for Ithaca. The value of the lines showing

¹ A clear day is one having on the average not more than three-tenths of the sky covered by clouds; a partly cloudy day is one having on the average from four to seven-tenths (inclusive) of the sky covered by clouds; and a cloudy day is one on which the sky is overcast or at least eight-tenths covered by clouds.—U. S. Weather Bureau.

the distribution of sunshine is therefore less than if all the elements entering into the sunshine could be combined into a single curve.

Chart No. 2 shows the curve for the sugar in the beet, the purity of the juice, the temperature, and the average length of the day. In this chart we have a remarkable illustration of the influence of high temperature upon sugar content. The two curves make almost an X-shaped figure. Low sugar and high temperature evidently go together. The highest temperature record for the summer was at Raleigh, N. C., and the lowest at Agricultural College, Mich. The temperature curve could also be very profitably compared with the latitude curve on Chart No. 1. It would form, also, an X-like figure with that curve. The purity of the juice shows a general tendency to follow the percentage of sugar, though there are many variations from this rule. In general, however, it is shown that the higher the percentage of sugar the higher the purity.

The curve showing the average length of day from sunrise to sunset has a direct relation also to the content of sugar in the beet. The shorter the day the lower the content of sugar and the longer the day the higher the content of sugar. This variation is doubtless partly due to the longer action of the sun's rays either directly or diffused through the clouds upon the sugar producing cells of the beet.

In Chart No. 3 are platted the curves showing the percentage of sugar in the beet, the total rainfall, the distribution of rainfall by months, and the altitude of the station above the sea level. The general influence of altitude, as is well known, is to lower the temperature for a given latitude. In other words, the altitude to a certain extent becomes a function of the latitude curve, and it would probably be advisable in some way to combine the two into a single curve. The highest altitude of the experimental stations collaborating (exclusive of Logan, Utah, which is not included in the charts) is at Lexington, Ky., namely, 979 feet; the lowest (sea level) at Washington. Other notably high stations are at Ames, Iowa, and Madison, Wis., and other notably low stations are at Raleigh, N. C., and Geneva, N. Y.

A more important relation to sugar content is shown by the rainfall, especially in its distribution. The total amount of rainfall, it is evident, has less influence on the sugar content than its even distribution during the growing months, providing the rain is sufficient for the growing crop. The greatest rainfall shown by any of the stations was at Ames, Iowa, and the lowest at Ithaca, N. Y. The rain at Ames was evidently far in excess of the requirements of the growing crop. The Washington rainfall was quite sufficient in quantity, but it was extremely uneven in distribution. For instance, during the month of June about 11 inches of water fell in Washington, while in July and August, when water was most needed, the amount was only about 1 and 2 inches, respectively. A very even distribution of rainfall is shown in the station at Geneva,

N. Y., while the quantity was relatively small. The distribution of the rainfall, also, at Ithaca was somewhat even, but there was a slight excess in October at a time when it would be injurious to the beet in the way of inducing a second growth. On the other hand, the September rainfall at Ithaca was small, thus favoring the ripening of the beet. The ideal conditions for the growth of the beet are an even distribution of the rainfall of from 3 to 4 inches during the months of May, June, July, and August, and a reduction of the rainfall for September and October.

The above conclusions, derived from these studies of a year, are quite in harmony with the theories which already prevail in regard to the effect of seasonal influences upon the sugar content of the beet. There are many problems, however, presented by the data which offer an inviting field of study. Chief among these is the suggestion, which has already been made in a previous part of this bulletin, that the high temperature line which seems to be so disastrous in its effects upon the sugar content of the beet may not produce all these ill effects directly as the result of the high temperature, but indirectly in the effect produced upon the moisture in the soil, the arrest of growth by dry weather, the inducement of a second growth on the accession of rains following a drought, and in other indirect ways. The study of this problem would best be carried on in an irrigated arid region where the temperature is high during the growing months and where the distribution of water on an experimental plat could be absolutely controlled. Other new problems of interest are also presented in studying the effects of direct and indirect sunshine and the distribution of the hours of direct sunshine compared with indirect and with partly cloudy weather.

In the study of these problems so far we are indebted to the cordial cooperation of the Weather Bureau and experiment stations, and in the further elaboration of them we rely on the promise of the continuance of this aid. It is certain that environment, of which meteorological conditions form the chief component, have a most marked influence on the chemical composition of crops, and without the assistance of the Weather Bureau it would be difficult to properly study the extent of the changes produced.

The analytical work in connection with these investigations was conducted by Dr. G. L. Spencer, in charge of the sugar laboratory of this Bureau, to whom I am indebted for many suggestions in the preparation of this bulletin.

U. S. DEPARTMENT OF AGRICULTURE.
BUREAU OF CHEMISTRY—BULLETIN NO. 65.

H. W. WILEY, Chief.

PROVISIONAL METHODS

FOR THE

ANALYSIS OF FOODS

ADOPTED BY THE

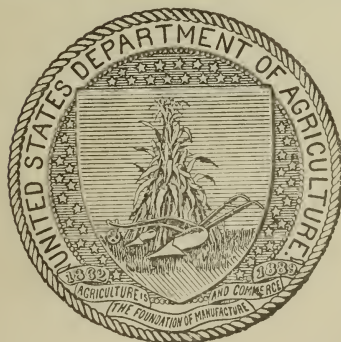
ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,
NOVEMBER 14-16, 1901.

EDITED BY

H. W. WILEY, SECRETARY,

WITH THE COLLABORATION OF

W. D. BIGELOW, REFEREE ON FOOD ADULTERATION.



WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1902.

LETTER OF TRANSMITTAL

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., January 7, 1902.

SIR: I have the honor to submit for your inspection and approval the manuscript of the Provisional Methods of the Association of Official Agricultural Chemists for the Analysis of Foods, with the recommendation that it be published as Bulletin No. 65 of the Bureau of Chemistry.

H. W. WILEY,
*Chief of the Bureau of Chemistry and Secretary of the
Association of Official Agricultural Chemists.*

Hon. JAMES WILSON,
Secretary of Agriculture.

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INTRODUCTION.

At the meeting of the association in 1900 it was decided, at the suggestion of Mr. Kilgore, the retiring president, to divide the subject of food adulteration into a number of general classes, and make a systematic effort to outline methods for their examination. With this in view, the referee in charge of this subject was instructed to secure the cooperation of associate referees, each of whom should prepare methods for the examination of one or more classes of foods. It was recognized that these methods could not all be prepared at once, but it was the desire of the association that a beginning be made, and that the work be prosecuted with as much vigor as possible. The work was immediately organized, and the cooperation of the following associates was secured: W. M. Allen, W. H. Ellis, William Frear, F. T. Harrison, A. E. Leach, J. A. Le Clerc, A. McGill, A. S. Mitchell, L. S. Munson, L. M. Tolman, H. W. Wiley, and A. L. Winton.

The reports, when completed, were forwarded to the referee, printed, and distributed to a mailing list of about 250 chemists for suggestions and criticisms, and a meeting of the entire committee was convened just before the meeting of the association in November, 1901. The methods as amended at this meeting were reported to the association and adopted provisionally. In several cases the reports which follow are the result of extensive work which was performed largely for the preparation of these methods. In other cases it has only been possible to take up a portion of the subject; and in still other instances it was found necessary to defer reports for another year.

On the whole, it may be said that the methods which were presented are more complete than was anticipated. Several who had not expected to make any report until the following year have been able to prepare a creditable outline of their subjects, and all the reports promised for this year have been received. It is considered, however, by those who have the matter in hand, that only a beginning has been made and that experience will indicate numerous changes in the methods which will be advantageous. At the same time the subject is placed in such a position that it may be considered in detail at an earlier date than was expected.

The writer desires to express his obligation to all of his associates, and especially to the local associates, Messrs. Munson and Tolman, for their cordial, prompt, and efficient cooperation.

W. D. BIGELOW,
Referee on Food Adulteration.

PROVISIONAL METHODS FOR THE ANALYSIS OF FOODS, ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, NOVEMBER 14-16, 1901.

I. MEAT AND MEAT PRODUCTS.

By W. D. BIGELOW,

In Charge of Food Laboratory, Bureau of Chemistry, U. S. Department of Agriculture.

A. MEAT.

1.—IDENTIFICATION OF SPECIES.

When dealing with large pieces of meat, and especially with fresh meat, the determination of the species of animal from which it was taken is the work of the veterinarian rather than the chemist, although the data obtained by the latter are often conclusive as to the variety of meat present. The physical appearance of the meat, its luster, grain, compactness, the presence or absence of the marbled appearance due to intermuscular fat, the size and shape of the bones, and the color and consistency of the fatty tissue must all be taken into account. Many of these characteristics are destroyed by curing or smoking, and none of them are retained by chopped meat, sausage, potted meat, or other preparations of like nature.

In such cases we must depend mainly on the results of chemical examination. The percentage of glycogen, added to the percentage of reducing sugar, is often of value in detecting horse meat in preparations which are supposed to consist of beef. Certain results obtained in the examination of fat separated from the meat by heat or by extraction with organic solvents also afford valuable data. Among the factors which are of value for this purpose may be mentioned the iodine number, melting point, freezing point, index of refraction, and to a less extent the specific gravity, acetyl number, and Maumené value.^a The meat from embryonic animals and from animals killed before they are suitable for food may often be detected by its moist, clammy nature and high water content.

2.—EXAMINATION OF POISONOUS MEAT AND OTHER FOODS.^b

From a hygienic standpoint the recognition of diseased meat is a matter of prime importance. The inspection of fresh meats for the purpose of detecting animal parasites such as trichinæ and vegetable parasites such as the lumpy-jaw fungus, the bacillus of tuberculosis, and other disease-producing bacteria, need not concern the analyst. It is only when a food, presumably wholesome, is found to have poisoned one or more individuals that the analyst must, to some degree at least, distinguish between the agents that may play a causative rôle.

The ordinary foods of man are liable to become poisonous from either of the three causes: (1) Trichinæ in pork, (2) metals, and (3) bacterial products.

^a See Appendix, p. 149.

^b The matter under this heading was written for this bulletin by Dr. F. G. Novy, of Ann Arbor, Mich.

(a) TRICHINÆ.

Pork, and sausage containing pork, which has caused sickness should be examined at once for trichinæ.^a

(b) POISONOUS METALS.

The poisonous metals arsenic, antimony, tin, lead, copper, and zinc are to be considered. It should be borne in mind, however, that food poisoning from metals is extremely rare compared with the causes mentioned under bacterial products. It is furthermore not uncommon to find minute amounts of tin or lead in canned meats or other canned food, and the accidental development of toxic properties in such canned goods can not stand in any causal relation to such minimal amount of metals. A single small dose of copper, lead, tin, or other metal need not in itself cause any unpleasant effect, but continued dosing with such small amounts may in the end give rise to disturbances. Several instances of this kind may be mentioned. Vaughan, some years ago, found the cattle in a Western mining region to be dying off because the stream from which they drank received the washings from a hydraulic mining camp. An investigation showed that both arsenic and antimony were present in solution as well as in suspension. Again, a garrison stationed on an East Indian island was obliged to use exclusively water stored up in a galvanized iron tank, with the result that chronic gastroenteritis developed in nearly every man. The experience with arsenic in beer in Manchester is very recent. Several hundred cases of chronic peripheral neuritis followed the continued use of beer containing minute amounts of this poison. On the other hand, acute poisoning from metals added intentionally or by mistake to foods are too well known. The amount of poison in such cases is such as to render the whole matter easy of solution.

From what has been said it is evident that poisonous meats could only under the most exceptional conditions owe this property to metals. A chemical examination, beyond revealing the merest traces of such metals as tin or lead, would mean nothing. More than that, such an examination requires a relatively large amount of material, and it not infrequently happens that the chemist hastens on with his chemical examination, to which he subjects all or nearly all of his material, so that when he attains a negative result scarcely any of the original substance is left for examination along other lines. For the determination of heavy metals, proceed as directed under vegetables (p. 52).

(c) BACTERIAL PRODUCTS.

The vast majority of all food poisonings are due to the invasion of the food by bacteria. The mere fact that bacteria are present in a meat is not evidence that such food is poisonous. Many, in fact most, of the bacteria which invade food are incapable of producing poisons, but they may grow and multiply and give rise to observable decomposition changes. Such changes can be spoken of as simple decomposition due to invasion by nonpoisonous bacteria. When, however, poison-producing germs develop in the meat, then as food it becomes poisonous. It is a noteworthy fact that a food may be highly poisonous without any visible indication of decomposition. In other words, odor and taste are not always reliable guides as to the innocuousness of a food.

The toxicogenic germ present in a food may be such that it can not grow in the body, and hence it is obvious that the poisonous effects which are observed are due to the poison which the germ had elaborated while growing in the meat. Such poisonings are pure intoxications. Another type of germ not only grows in the meat where it makes some poison, but it can also grow in the body, and as a result it continues to elaborate such poison for some time after being ingested. And, lastly, a germ may be present which can not grow at ordinary temperature, but does grow in the

^a Fischöder, Leitfaden der praktischen Fleischschau; Ostertag, Handbuch der Fleischschau; Walley, A Practical Guide to Meat Inspection.

body, in which case the poisoning is a true infection. Instances are known where diseases of animals have been transmitted by eating the flesh.

In view of the fact that bacteria are, above all, the most common cause of poisonous meat, it follows that the examination should primarily be made from that standpoint. To sum up what has already been said: Given a poisonous meat, the first procedure is to detect or exclude the presence of trichinae. If they are not found, the bacteriological examination should next be undertaken, and the chemical examination should be reserved until the last.

The bacteriological examination should first consist in feeding a number of different species of animals—the larger the number the better—for a day or two exclusively upon the food. White mice, house mice, white rats, young dogs, cats, rabbits, or guinea pigs can be used. If the animals sicken and die they are to be subsequently examined for the presence of pathogenic bacteria. It may happen that none of the animals thus fed will be injured by the food, which fact would not exclude, however, the presence of a germ requiring a specially susceptible animal for a subject.

Another set of animals should be injected with a cold extract of the meat made with sterile water. If the animals die, they are to be examined for pathogenic bacteria. A third set of animals should receive similar injections, though of larger portions, of this aqueous extract which has been previously filtered through sterile porcelain. If the animals die from such injections the same as with unfiltered solutions, it is evident that a soluble bacterial chemical poison is present.

The identification of the toxin or real poison produced by the germ is wholly out of the question. The most that can be done satisfactorily is to obtain,* as above, a germ-free solution of the poison. It is wholly unnecessary to devote any space in this connection to the detection of the basic bacterial products, the ptomaines, since these bodies are mere cleavage products produced by some and not by other bacteria. Moreover, they are usually but very feebly poisonous, and for that reason they do not hold the prominent position formerly ascribed to them^a.

A bacteriological examination proper should be made of the original poisonous meat and of all the animals that died either from eating the meat or from the injections of the aqueous extracts. The organism present in the animal, if any, must be isolable directly from the meat. If it happens, as it sometimes has, that the dead animals contain no germs, it is proof that they were killed by a toxin elaborated by a germ in the meat previous to the injection. Cultures from the meat will then reveal the germ, and the effects of its pure cultures should correspond to those observed with the poisonous meat.

To prepare the cultures from the original food, the latter should be cut out with a sterile knife and material should be taken from the inside, thus avoiding all chances of contamination. Several sets of beef-tea tubes and agar plates should be made. One set should be set aside in a Novy anaerobic jar at room temperature; a second similar set should be placed at a temperature of 37° C. A third set should be grown in the presence of air at room temperature, and a like set at a temperature of 37°.

The full details of bacteriological methods must obviously be omitted in this connection. Such work requires a special laboratory and special drill. Those who may be further interested are referred to the works of Abbott, Novy, and Sternberg.

3.—PREPARATION OF SAMPLE FOR CHEMICAL EXAMINATION.

In the case of fresh meat, separate the sample as completely as possible from the bones and pass it rapidly and repeatedly through a sausage mill until thorough mixture and complete maceration are obtained. The sample must be kept on ice to prevent decomposition, and all of the determinations should be begun as soon as practicable after the sample is prepared. In the case of canned meats, pass the

*For detailed methods see Vaughan and Novy, "Cellular Toxins," 4th edition, 1902.

entire contents of a can through a sausage mill as directed above. Remove sausage from the casings and mix by repeated grinding in a sausage mill. Dry that portion of the sample which is not needed for analysis, extract with gasoline which boils below 60° C, allow the gasoline to evaporate spontaneously, and expel the last traces by heating for a short time on the steam bath. Neither the meat nor the separated fat should be heated longer than necessary, owing to the tendency of the latter to decompose. Reserve the fat for examination according to the methods given under the examination of edible fats and oils (page 20). Fat must be kept in a cool place, and its examination finished before it becomes rancid.

4.—DETERMINATION OF WATER.^a

Dry to constant weight about 2 grams of the macerated sample, in a tared, flat-bottomed dish at the temperature of boiling water. The dish may be of aluminum or platinum, or a tin bottle cap answers admirably for this purpose. On account of the oxidation of the fat, meats may be dried to advantage in a current of hydrogen or *in vacuo*, although satisfactory results are obtained in the above way. Drying usually requires about five hours.

5.—DETERMINATION OF ASH.²

Ignite the residue from the determination of water to low redness as long as smoke or inflammable gases are given off. Exhaust the charred mass with 5 or 10 cc of water, transfer to a filter, and wash with hot water till the greater part of the soluble salts are removed. Transfer filter paper and contents to the original dish and ignite at bright red heat till combustion is complete (a white ash can rarely be obtained). Transfer the soluble portion to the dish, add a few drops of ammonium-carbonate solution, evaporate to dryness, heat for a moment in a free flame to very low redness, cool in a desiccator and weigh. Satisfactory results may often be obtained without exhaustion by igniting 0.5 gram of the substance in a porcelain crucible cover.

6.—DETERMINATION OF ETHER EXTRACT.

It has recently been shown that fat can not be completely extracted from meat by means of ether. A complete extraction can be obtained only after digesting the proteids and muscular tissue with pepsin and extracting again with an organic solvent. Voit^b extracts first with alcohol, to remove the last traces of water, and then with ether in a continuous extractor. This process leaves very little fat in the sample. Comparative results which are satisfactory in all ordinary examinations of meat may be obtained by extracting 2 grams of the dried, finely divided sample with ether for 16 hours in a continuous extractor. Fat may be determined by extracting the ether extract with low boiling-point petroleum ether.

7.—DETERMINATION OF NITROGENOUS SUBSTANCES.

(a) TOTAL NITROGEN.

Employ either the Kjeldahl or the Gunning method, using about 2 grams of the sample. The digestion with sulphuric acid should be continued at least 4 hours.

(b) COAGULATED PROTEIDS.

Thoroughly exhaust 2 grams of the sample with cold water after extraction with ether, filter, and determine nitrogen in the insoluble residue as directed under "Total nitrogen." Multiply the percentage of nitrogen so obtained by 6.25 for the percent-

^a See Appendix, p. 149.

^b Ztschr. f. Biol., 1897, 35, 555.

age of meat fiber or coagulated proteids. (In case the connective tissue is determined, a corresponding correction must be made in the percentage of coagulated proteids.)

(c) DETERMINATION OF CONNECTIVE TISSUE.

Extract 10 grams of the sample with cold water as directed above, then boil the exhausted residue repeatedly with about 100 cc of water until the total extract amounts to about 1 liter. Filter the extract, concentrate by evaporation, and determine the nitrogen content. Multiply the nitrogen so obtained by 5.55 for the percentage of nitrogenous substances of connective tissue.

(d) DETERMINATION OF COAGULABLE PROTEIDS (FOR UNCOOKED MEAT ONLY).

Almost neutralize the filtrate from the coagulated proteids, leaving it still faintly acid, boil until the globulins are coagulated, filter, wash, transfer the filter paper and contents to a Kjeldahl flask, and determine nitrogen as directed above under "Total nitrogen." Multiply the percentage of nitrogen obtained by 6.25 for the percentage of coagulable proteids.

(e) DETERMINATION OF PROTEOSES, PEPTONES, AND GELATIN.

(1) *First method.*

This is a combination of Bömer's^a method with that of Allen and Searle,^b as modified by Wiley.^c

Evaporate the filtrate from the globulins to small volume, add 2 or 3 drops of 1-3 sulphuric acid, and saturate with powdered zinc sulphate. The excess of zinc sulphate added should not be large, as otherwise serious "bumping" is likely to ensue. About 80 grams of the salt are required for each 50 cc of liquid. Allow the coagulated proteids to subside, filter, and wash with a saturated solution of zinc sulphate.

Acidulate the filtrate from the zinc sulphate precipitate with 2 or 3 drops of strong hydrochloric acid, dilute with an equal volume of water, add about 2 cc of liquid bromin, and shake the contents of the flask vigorously. (This can be most conveniently done in a Kjeldahl flask.) If the bromin be all taken up, add more until about 0.5 cc of liquid bromin is left undissolved and the supernatant liquid thoroughly saturated. Allow the mixture to stand over night, decant the supernatant liquid through a filter paper, and wash with water, so directing the jet that the globule of bromin is stirred up and saturates the wash water. Return the filter paper and precipitate to the flask, add the zinc sulphate precipitate and filter paper containing it, and determine the nitrogen as directed under "Total nitrogen." The percentage of nitrogen so found, multiplied by 6.25, gives the percentage of proteoses, peptones, and gelatin, including gelatin peptone.

(2) *Second method.*^d

Heat the filtrate from albumen and globulins, add a slight excess of tannic acid and a few drops of a saturated solution of alum, allow to cool, filter, and wash with cold water. Heat the filtrate from the tannic-acid precipitate almost to boiling, add an excess of phospho-tungstic acid,^e separate the precipitated proteids by filtration

^a Ztschr. anal. Chem., 1895, 5, 562.

^b The Analyst, 1897, 22, 258-263.

^c U. S. Dept. Agr., Div. of Chem., Bul. 54.

^d Mallet, U. S. Dept. of Agr., Div. of Chem., Bul. 54.

^e Mallet employs two solutions, one containing 50 and the other 100 grams of crystalline phospho-duodeci-tungstic acid dissolved in 1 liter of 2½ per cent of hydrochloric acid. He also recommends the addition of sand or pulverized glass to prevent the formation of the coagulated proteids in a dense clot. Owing to the liability of "bumping" in the presence of such substances, however, during the determination of nitrogen it would seem that such addition should be avoided if possible.

and wash with hot water, being careful that the temperature of the solution and wash water shall not be less than 90° C. at any time.

Transfer the filter papers containing the tannic acid and phospho-tungstic acid precipitates to a Kjeldahl flask and determine nitrogen. The nitrogen so obtained multiplied by 6.25 gives the percentage of proteoses, peptones, and gelatin.

(f) DETERMINATION OF MEAT BASES.

Deduct from the total nitrogen the sum of the nitrogen obtained in the determination of coagulated proteids, connective tissue, globulin, and proteoses, peptones, and gelatin for the nitrogen of meat bases. Multiply the result by 3.12 for the percentage of meat bases.^a

8.—DETERMINATION OF STARCH (FOR CHOPPED MEAT, SAUSAGE, DEVILED MEAT, ETC.).

(a) QUALITATIVE DETERMINATION.

Treat 5 or 6 grams of the sample with boiling water for two or three minutes; cool the mixture, and test the supernatant liquid with iodine solution. In using this test it must be remembered that a small amount of starch may be present as the result of the use of spices. If a marked reaction is given, however, it may be concluded that starch or flour has been added, and a quantitative determination may be made. The above qualitative method may be replaced by a microscopic examination, by which not only the presence of added starch, but also the variety employed, may be determined.

(b) QUANTITATIVE DETERMINATION.

The official methods of the association for the determination of starch will not answer for the examination of meat because of the presence in meat of bodies which hold a portion of the cuprous oxid in solution, and thus give results which are too low. In this laboratory Munson examined a series of sausages which contained no starchy material except the spices employed in their manufacture. He obtained less reduced copper from the sausages than from the blank determination of reducing bodies in the malt infusion employed.

(1) *Ambühl's method.*^b

This method has been adopted by Swiss authorities. It is short and convenient, although the results obtained by it are only roughly approximate.

Thoroughly macerate 2 grams^c of the meat under examination with fifty times its weight of water. Boil for thirty minutes and dilute to 100 cc for every gram of meat employed. Cool an aliquot portion of the liquid, treat with iodine, and compare the depth of color with solutions containing a known amount of the same kind of starch (the variety of starch in the sample is determined microscopically), and boiled for the same length of time.

(2) *Mayrhofer's method,*^d *modified by Bigelow.*^e

Treat from 10 to 20 grams of the sample under examination (depending upon the amount of starch indicated by the iodine reaction) in a porcelain dish or casserole with 50 cc of an 8 per cent solution of potassium hydroxid and heat the mixture on the water bath until the meat is entirely dissolved. The operation may be hastened by macerating the larger pieces with a glass rod. Add an equal volume of 95 per

^a See appendix, p. 149.

^b Pharm. Centralh., 1881, 22, 438; Abstract Ztschr. anal. Chem., 1882, 21, 436.

^c Ambühl directs that from 2 to 10 grams be employed, according to the size of the meat particles. If the sample be macerated, however, as directed under the preparation of sample, it is unnecessary to employ a large amount.

^d Forsch. ü. Lebensm., 1896, 3, 141, and 1897, 4, 47.

^e U. S. Dept. of Agr., Bureau of Chem. Bul. 13, part 10.

cent (by volume) alcohol, mix thoroughly, filter the mixture through an asbestos filter and wash twice with a hot 4 per cent solution of potassium hydroxid in 50 per cent alcohol. Then wash with 50 per cent alcohol until a small portion of the filtrate does not become turbid upon the addition of acid. Return the precipitate and filter to the original vessel and dissolve the precipitate, with the aid of heat, in 60 cc of a normal solution of potassium hydroxid. In the case of sausage with a high starch content a somewhat larger volume of alkali may be required. Acidify the filtrate strongly with acetic acid, dilute to a definite volume, thoroughly mix by shaking, filter through a fluted filter, and precipitate the starch from an aliquot part of the filtrate by means of an equal volume of 95 per cent alcohol (sp. gr. 0.81). Transfer the precipitate to a filter, thoroughly wash with 50 per cent alcohol (by volume), with absolute alcohol, and finally with ether, dry to a constant weight at the temperature of boiling water, and weigh.

9.—DETERMINATION OF GLYCOGEN.

The determination of glycogen has been suggested^a as a means to detect the presence of horse meat. Recent results indicate that this determination is of limited value because of the fact that glycogen begins to disappear soon after the death of the animal and may entirely disappear after a short lapse of time. No definite conclusions can therefore be derived from the results of this determination, but it is of value as confirmatory.

(a) QUALITATIVE METHOD.^b

Boil 50 grams of the macerated sample with 50 cc of water for from fifteen to thirty minutes. Filter the broth through a moistened filter paper or piece of fine linen. To a portion of the filtrate in a test tube add a few drops of a reagent composed of 2 parts iodine, 4 parts potassium iodide, and 100 parts water. In the presence of a large percentage of horse meat the glycogen contained produces a dark brown color, which is destroyed by heating and reappears on cooling. When starch is present it may be precipitated by two volumes of glacial acetic acid, separated by filtration, and the test for glycogen repeated in the filtrate.

(b) QUANTITATIVE METHOD.

The methods for the quantitative examination of glycogen are all tedious to the last degree. Fairly satisfactory results may be obtained by the methods of Brücke,^c R. Külz,^d Pflüger,^e Haywood,^f and Pflüger and Nerking.^g Of these the last has been selected as combining a sufficient degree of accuracy with the greatest simplicity and convenience.

Digest 50 grams of finely macerated meat on the water bath with 200 cc of 2 per cent potassium hydroxid until solution is practically complete. Cool the solution, dilute with water to exactly 200 cc, shake and filter. Treat 100 cc of the filtrate with 10 grams of potassium iodide and 1 gram of potassium hydroxid and stir until solution is complete. Add 50 cc of 96 per cent alcohol, and allow to stand until the following day. Then separate the precipitated glycogen by filtration, and wash with a solution containing 1 cc of 73 per cent potassium hydroxid, 10 grams of potassium iodide, 100 cc of water, and 50 cc of 96 per cent alcohol (sp. gr. .81). Wash the glycogen with a mixture of two volumes of 96 per cent alcohol and one volume of water containing about 7 mg of sodium chlorid per liter, dissolve in water, and remove the remaining traces of proteids by the addition of double iodide of mercury and potassium. It is often

^a Niebel. *Ztschr. ang. Chem.*, 1895, 620.

^b Courlay and Coremons. *Ztschr. Nahr. Hyg. Waar.*, 1896, **10**, 173-174.

^c *Sitzungsber Acad. Wissensch., Wien*, Bd. 63, II abth., 1871, p. 214.

^d *Ztschr. f. Biol.*, **1**, 169.

^e *Arch. ges. Physiol.*, 1899, **75**, 120-247.

^f *Jour. Am. Chem. Soc.*, 1900, **22**, 85.

^g *Arch. Physiol.*, 1899, **76**, 531-542.

found that the proteids are so completely removed that no precipitate is formed with the double iodid. In such case filtration is not necessary.

Add about 2 mg of sodium chlorid per 100 cc of water, precipitate the glycogen again by means of two volumes of 96 per cent (sp. gr. 0.81) alcohol, filter, wash with 96 per cent alcohol, containing about 7 mg of sodium chlorid per liter, then with absolute alcohol, finally with ether, dry to constant weight, and weigh.

As a control, invert the precipitated glycogen by boiling for three hours with hydrochloric acid diluted with 10 parts of water and determine the reducing sugar by Allihn's method, multiplying the result by 0.9 for percentage of glycogen.

10.—DETERMINATION OF REDUCING SUGAR.

Boil 100 grams of the finely divided meat for fifteen or twenty minutes in a 500-cc graduated flask with a convenient volume of water. Add a few cubic centimeters of normal lead acetate, cool to room temperature, make up to mark with water, and filter through a fluted filter. Evaporate to a small volume as large an aliquot portion of the filtrate as possible, add a saturated solution of sodium sulphate, make up to a definite volume, and filter through a fluted filter. Determine sugar in an aliquot portion of the filtrate by the Allihn method (p. 49).

11.—DETERMINATION OF POTASSIUM NITRATE.

(a) METHOD OF SCHLÖSING-WAGNER.^a

A flask of about 250-cc capacity is provided with a rubber stopper with two holes. Through one of them is passed the stem of a funnel carrying a glass stopcock. The other carries a delivery tube leading to the receiving vessel. The end of the delivery tube is bent so as to pass easily under the mouth of the measuring burette and is covered with a piece of rubber tubing.

Fifty cubic centimeters of saturated ferrous chlorid solution and the same quantity of 10 per cent hydrochloric acid are placed in a flask. The ferrous chlorid solution is prepared by dissolving nails or other small pieces of iron in hot hydrochloric acid and is kept in glass-stoppered flasks of about 50-cc capacity, entirely filled. The contents of one flask is enough for about twelve determinations, and by using the whole content of a flask as soon as possible after opening, all danger of oxidation which would take place in a large flask frequently opened is avoided.

The contents of the flask are boiled until all the air is driven off. The delivery tube is then placed under the measuring tube, which is filled with 40 per cent potassium hydroxid, then a few drops of water are added and the tube is covered with a piece of filter paper. By a careful and quick inversion, the measuring tube can be brought into the vessel receiving it without any danger of air entering.

One hundred grams of the finely macerated meat are extracted by boiling repeatedly with successive small volumes of water, the aqueous solution is concentrated to a small volume, transferred to the funnel, and, with continued boiling, allowed to pass, drop by drop, into the flask. When almost all has run out, the funnel is washed with three 10-cc portions of 10 per cent hydrochloric acid and these portions are allowed to pass, drop by drop, into the flask; the temperature of the surrounding water will soon be imparted to the contents of the tube, and the volume of nitric oxid is read with the tube in such a position that the level of the water within and without the tube coincide.

The amount of nitric oxid present and the corresponding percentage of nitrate may be calculated in the usual way for the given temperature and barometric pressure, or, to avoid computation, the amount of nitrate may be determined by comparison of the

^aAgr. Chem. Vers. Stat. Halle, p. 50; Wiley, Principles and Practice of Agricultural Analysis, vol. 2, p. 228.

volume of nitric oxid with that evolved by a definite volume (5 to 10 cc) of normal sodium nitrate solution.

(b) PHENOL-SULPHONIC ACID METHOD.^a

Weigh 1 gram of the sample into a 100-cc flask, add from 20 to 30 cc of water, and heat on the water bath for fifteen or twenty minutes, shaking occasionally. Add 3 cc of a saturated solution of silver sulphate for each per cent of sodium chlorid present, then add 10 cc of lead subacetate and 5 cc of alumina cream, shaking after each addition. Make up to mark with water, and filter through a fluted filter, returning the filtrate to the filter until it runs clear. Evaporate to dryness 25 cc of the filtrate, add 1 cc of phenol-sulphonic acid,^b mix thoroughly with a glass rod, add 1 cc of water and 3 or 4 drops of concentrated sulphuric acid and heat on a steam bath for two or three minutes, being careful not to raise the temperature sufficiently to char the material. Now add about 25 cc of water and an excess of ammonium hydroxid. Transfer to a 100-cc flask, add 1 or 2 cc alumina cream if not perfectly clear, dilute to mark with water, and filter if necessary.

Prepare a number of 50-cc Nessler tubes, preferably the long, narrow tubes, placing in the first 1 cc of the standard nitrate solution, in the second 2 cc, and so on to 10 cc, then 12 cc, 15 cc, 18 cc, and 20 cc. The comparison of the solution under examination with these tubes will show directly if it comes within this range, in which case it can be read by direct comparison with the various tubes till the one of the exact shade is found. If the color of the solution be darker than any of the tubes prepared as above, it is preferable to dilute as many times as may be necessary to bring the color within this range by transferring 25 cc of the solution to another tube with a pipette and filling up to the mark with distilled water. In this case the reading of the diluted solution in cubic centimeters of standard solution should be multiplied by the number of times the solution under comparison has been diluted. More exact comparisons can be made looking sidewise through the tubes toward a window covered with white paper and shaded from direct sunlight.

The following table prepared by Mr. Given enables one to determine at a glance the percentage of potassium nitrate in a given sample from the number of cubic centimeters of standard solution employed, if the above directions are followed in detail:

Per cent potassium nitrate.

Stand- ard solu- tion.	Per cent KNO ₃ .	Stand- ard solu- tion.	Per cent KNO ₃ .	Stand- ard solu- tion.	Per cent KNO ₃ .
cc		cc		cc	
0.7	0.01	14.7	0.21	28.7	0.41
1.4	.02	15.4	.22	29.4	.42
2.1	.03	16.1	.23	30.1	.43
2.8	.04	16.8	.24	30.8	.44
3.5	.05	17.5	.25	31.5	.45
4.2	.06	18.2	.26	32.2	.46
4.9	.07	18.9	.27	32.9	.47
5.6	.08	19.6	.28	33.6	.48
6.3	.09	20.3	.29	34.3	.49
7.0	.10	21.0	.30	35.0	.50
7.7	.11	21.7	.31	35.7	.51
8.4	.12	22.4	.32	36.4	.52
9.1	.13	23.1	.33	37.1	.53
9.8	.14	23.8	.34	37.8	.54
10.5	.15	24.5	.35	38.5	.55
11.2	.16	25.2	.36	39.2	.56
11.9	.17	25.9	.37	39.9	.57
12.6	.18	26.6	.38	40.6	.58
13.3	.19	27.3	.39	41.3	.59
14.0	.20	28.0	.40	42.0	.60

^a This method is a modification of the one ordinarily employed for determining potassium nitrate in water. It was adapted to the examination of meat by Mr. Arthur Given.

^b Prepared by mixing together 37 cc of concentrated sulphuric acid, 3 cc of distilled water, and 6 grams of phenol.

12. DETECTION OF PRESERVATIVES.

The chemical preservatives commonly used with meat products are borax and boric acid and sulphites. Salicylic and benzoic acids are occasionally used, and formaldehyde is said to be used, although the writer has failed to detect its presence in meat preparations. The general methods for the detection of these preservatives are given on pages 107 and 110. A few special methods are described below. In general, preservatives may be separated from meat by digesting a few minutes in warm water, made slightly acid or slightly alkaline according as the nature of the preservative is basic or acid.

(a) BORAX AND BORIC ACID.

If present in noticeable amounts, boric acid may be detected in meat products by heating 20 grams of the sample a few minutes in about 100 cc water acidified with 6 or 8 cc of concentrated hydrochloric acid and testing with turmeric paper as directed on page 110. If no action is obtained by this method, about 20 grams of the sample should be made alkaline with calcium hydroxid, ignited, and the ash tested as directed under preservatives.

(b) SULPHUROUS ACID.

The distillation method for the detection of sulphurous acid (see page 107) will answer for the examination of meat, but mere traces should be ignored. According to Ostertag,^a the microscopic examination of meat that has been preserved with sodium or calcium sulphite often discloses the presence of crystals of sodium or calcium sulphate, due to partial oxidation of the sulphite.

In the absence of chlorids and nitrates Kämmerer^a employs potassium iodate paper in the following manner: Place the sample of meat on potassium iodate paper and moisten it with dilute sulphuric acid (1:8) free from oxids of nitrogen. In the presence of even minute traces of sulphites a deep-blue color is immediately formed, while in the absence of sulphites only a faint-blue color is produced, and that after a considerable time. This method is of limited application, since it can not be used with meats containing salt or saltpeter.

13. DETECTION OF COLORING MATTER.^b

Sausages and other preparations in which chopped meat is employed rapidly become discolored on exposure to the air. This change does not take place to a marked extent with meat that has been cured in a pickle containing saltpeter. With fresh chopped meat, and sometimes with corned meat, especially that cured without saltpeter, coloring matter is sometimes added to prevent the change of color which would naturally take place. Aniline dyes and cochineal carmine are ordinarily employed for this purpose, though in some instances vegetable colors have been detected in the form of lakes. The coloring matter may often be extracted by heating for 15 or 20 minutes with 50 per cent alcohol, 50 per cent glycerin slightly acidified, a mixture of alcohol and glycerin,^c ammonium hydroxid, or a 5 per cent solution of sodium salicylate^d in water. Approximately equal weights of meat and solvent may be used.

In case the filtered extract by any of these methods is colored red or deep yellow, it should be evaporated nearly to dryness, slightly acidified with hydrochloric acid, and boiled a few minutes after the addition of a thread of fat free wool. If the wool is dyed, it may be examined as directed by the referee on coloring matter. If the wool is not dyed, the solution is examined spectroscopically.

^a Handbuch der Fleischbeschau, 3 ed., p. 826.

^b See appendix, p. 149.

^c Klinger and Bujard, Ztschr. ang. Chem., 1891, 515.

^d Spaeth, Pharm. Centralh., 1897, 38, 884.

If too dilute, the solution may often be concentrated by precipitating the coloring matter as a lake,^a allowing it to settle, decanting off the water, dissolving in hydrochloric acid and making alkaline with ammonia.

In extracting with 50 per cent alcohol, the proteids of the meat are coagulated, with the formation of a pale, almost white, color. If the meat is not discolored during this extraction, it is probable that some foreign color is present.^b

Marpmann^b examines sausages microscopically for the presence of coloring matter after dehydrating with alcohol and xylol consecutively, removing the xylol with carbon tetrachlorid, and immersing in cedar oil until the natural colors of the meat have disappeared.

(B) MEAT EXTRACTS.

1.—PREPARATION OF SAMPLE.

Liquid and semiliquid meat extracts and similar preparations should be removed from the container and thoroughly mixed before sampling. With many liquid preparations a sediment is found in the bottom of the container which will be overlooked if great care is not taken.

2.—DETERMINATION OF WATER.

Follow directions given on page 10, employing about 2 grams of powdered preparations, about 3 grams of preparations of pasty consistency, and from 5 to 10 grams of liquid extracts, according to the solid contents. Dry the powdered preparations directly without admixture. Dissolve the pasty preparations in water and dry with sufficient ignited asbestos or pumice stone to absorb the solution. Tin or lead dishes or Hofmeister glass dishes, are often convenient with samples in which the residue is to be extracted for fat, as the dishes may be cut or broken and placed in the extraction tube with the sample.

3.—DETERMINATION OF ASH.

Proceed as directed on page 10. In case of pasty preparations, add sufficient water to effect solution and evaporate to dryness in order that the solids may be distributed evenly over the bottom of the dish.

4.—DETERMINATION OF FAT.

Transfer the residue from the determination of water to the tube of a continuous extraction apparatus, wash any fat adhering to the dish into the tube with ether, and extract with ether sixteen hours.

5.—DETERMINATION OF NITROGENOUS SUBSTANCES.

(a) TOTAL NITROGEN.

Employ either the Kjeldahl or the Gunning method.

(b) DETERMINATION OF MEAT FIBER.^c

Dissolve in cold water 5 grams of powdered preparations, from 8 to 10 grams of extracts of pasty consistency, or from 20 to 25 grams of fluid extracts; filter and wash with cold water. Transfer the filter paper and contents to a Kjeldahl flask and determine nitrogen as directed under total nitrogen. In case of a large amount of insoluble matter, make up to a definite volume, filter through a fluted filter paper,

^aBremer, *Forschungsber.*, 1897, 4, 45.

^b*Ztschr. ang. Mikr.*, 1895, 1, 12.

^cAllen, *Com. Org. Anal.*, 2d ed., vol. 4, p. 324.

and determine nitrogen in an aliquot portion of the filtrate; then deduct the percentage of nitrogen in the total filtrate from the percentage of total nitrogen for the percentage of nitrogen in meat fiber. Multiply the percentage of nitrogen by 6.25 for the percentage of meat fiber.

(c) DETERMINATION OF COAGULABLE PROTEIDS.

Make the filtrate (as large an aliquot portion as practicable when the nitrogen of meat fiber has been determined by difference) from meat fiber slightly acid (if not already acid), adding acetic acid or sodium hydroxid as may be required, boil for two or three minutes, cool to room temperature, dilute to 500 cc and filter through a fluted filter.^a

Determine nitrogen in 50 cc of the filtrate by means of the Kjeldahl or Gunning method. Ten times the nitrogen so obtained deducted from the percentage of soluble nitrogen (which in turn is obtained by deducting percentage of nitrogen occurring as meat fiber from the total nitrogen) gives the percentage of nitrogen contained in albumin and globulins. Multiply this figure by 6.25 for the percentage of coagulable proteids in the sample.

(d) DETERMINATION OF SYNTONIN.

Exactly neutralize the filtrate from the determination of coagulable proteids with sodium hydroxid, using litmus as indicator, and allow to stand until the precipitate settles. If only a small amount of syntonin is precipitated, it may be separated with an ordinary filter, washed with water, and its nitrogen content determined by means of the Kjeldahl or Gunning method. If present in any considerable quantity, dilute to a definite volume, filter through a fluted filter, and determine nitrogen in 50 cc of the filtrate.

The nitrogen thus obtained (calculated to total volume) is deducted from the nitrogen in the filtrate from the globulins for the syntonin nitrogen. This multiplied by 6.25 gives syntonin.

(e) DETERMINATION OF PROTEOSES, PEPTONES, AND GELATIN.^b

If it be desired to group these bodies together, proceed as directed under (e), page 11, unite the two precipitates and make a single determination of nitrogen. The percentage of these bodies can not be determined by using aliquot parts and deducting the nitrogen content of the filtrate from the bromin precipitate from that of the filtrate from the determination of syntonin, because of the decomposing effect exerted by bromin on nitrogen compounds. Experiments in this laboratory also indicate that the aliquot portions of the filtrate from the determination of syntonin can not be used separately for the zinc-sulphate precipitate and the bromin precipitate for the same reason. Although bromin precipitates peptones and zinc sulphate does not, Trescot found, in the examination of a large number of meat extracts when working with aliquot portions of the same solution, that more nitrogen was precipitated by zinc sulphate than by bromin.

(f) DETERMINATION OF PROTEOSES AND GELATIN.^c

Evaporate the filtrate from the determination of syntonin (as large an aliquot portion of the filtrate as is practicable when the percentage of syntonin is determined

^aThe filtering and washing of coagulated proteids are always tedious and unsatisfactory and sometimes almost impossible. The work is greatly simplified, therefore, by passing through a fluted filter and employing aliquot parts of the filtrate, as by this means the complete filtration and washing of precipitates is made unnecessary.

^bAllen, *The Analyst*, 1897, **22**, 258; *Com. Org. anal.*, 2d edition, vol. 4, p. 325.

^cBömer, *Ztschr. anal. Chem.*, 1895, **5**, 562; also Mallet, U. S. Dept. Agr., Div. of Chem., Bul. 54.

by difference, as suggested by the writer) to a small volume and saturate with zinc sulphate. About 85 grams of powdered zinc sulphate are necessary for the saturation of 50 cc of the liquid at ordinary laboratory temperature. The liquid must be fully saturated with the salt, but a large excess should be avoided, as it is likely to cause "bumping" in the subsequent determination of nitrogen in the solution. Let stand several hours, filter, and wash the precipitate with saturated zinc sulphate. In case the precipitate is voluminous, which rarely happens, the mixture may be made up to a definite volume with saturated zinc sulphate, filtered, the nitrogen may be determined in an aliquot portion of the filtrate, and the nitrogen of the precipitated proteids determined by difference.

(g) DETERMINATION OF PEPTONES.^a

Dilute the filtrate from the zinc-sulphate precipitate of proteoses and gelatin with an equal volume of water, add bromin until a globule of from 0.5 cc to 1 cc remains undissolved after the liquid is saturated, and allow to stand over night. Filter, wash with cold water, directing the jet to the globule of bromin so as to keep the wash water saturated. Transfer the filtered precipitate to a Kjeldahl flask and determine nitrogen.

(h) DETERMINATION OF GELATIN.

Stutzer's method^b modified by Bigelow.^c

Boil 10 grams of the sample for a few minutes with water; filter, wash, and evaporate the filtrate to dryness in a porcelain dish of about 10 cm diameter, after the addition of about 20 grams of sand which has been freed from dust by sifting, and thoroughly ignited. Exhaust the residue with four 100-cc portions of absolute alcohol, and pass the supernatant liquid through an asbestos filter which rests on a porous plate of about 4 cm diameter, in a funnel. The funnel is surrounded by pounded ice and attached to an aspirator, by means of which gentle and gradually increasing suction may be applied. Take care to transfer as little as possible of the insoluble residue to the filter. Then extract the residue repeatedly with 100-cc portions of a mixture containing 100 cc of 95 per cent alcohol (sp. gr. 0.81), 300 grams of ice, and 600 grams of cold water, taking care that the temperature shall not exceed 5° C. at any time. Continue the extraction until the various portions of solvent used are entirely colorless. Filter the extract through the funnel employed for the alcohol extract. Finally, return the asbestos filter to the beaker which contains the exhausted residue and thoroughly extract the whole with boiling water. Receive the hot-water extract in a Kjeldahl flask, determine nitrogen, and multiply the percentage of nitrogen so obtained by 5.55 for the percentage of gelatin and gelatin peptone.

(i) PROTEOSES.

Deduct the nitrogen in the gelatin precipitate (h) from that of the proteose and gelatin precipitate. This multiplied by 6.25 gives the percentage of proteoses.

(j) MEAT BASES.

Deduct from the total nitrogen (a) the sum of the nitrogen in (b), (c), (d), (f), and (g). Multiply the difference by 3.12 for meat bases.

6.—DETERMINATION OF GLYCOGEN.

Proceed as directed on page 13.

7.—DETECTION OF PRESERVATIVES.

Proceed as directed under Preservatives, page 107.

^a Allen Com. Org. anal, 2nd Ed., vol. 4, p. 320.

^b Ztschr. anal. Chem., 1895, 34, 568.

^c U. S. Dept. of Agr., Bureau of Chem., Bul. 13, Part 10.

II. EDIBLE OILS AND FATS.

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1.—GENERAL DISCUSSION.

In working with oils and fats the same methods of examination largely apply, except in preparation of the sample.

The solid fats should first be melted, thoroughly mixed, and then filtered by means of a hot-water funnel or similar apparatus. Samples for the different determinations are taken from this melted homogeneous mass. The specific gravity must be taken at some temperature above the melting point of the fat. The boiling point of water has been largely used, and, although there are inherent errors in such a method,^a it probably gives the most satisfactory results for practical work.

In the Maumené test, fats require a higher initial temperature than oils.

With oils in most cases the sample for analysis requires no preliminary treatment, except that in case of impurities the oil should be filtered.

Oil and fat should always be kept in a cool place, otherwise they will soon become rancid, which will affect more or less the physical and chemical constants.^b The iodine number decreases with rancidity, while specific gravity, index of refraction, and acetyl value increase. Too much confidence must not be placed in negative results obtained in a single determination, but only upon making a complete quantitative as well as qualitative examination can a reliable judgment of purity be made.

Oils and fats being variable mixtures of glycerids of the fatty acids, their physical and chemical constants vary within limits fairly well established from analytical data. However, too much dependence must not be placed upon the more common determinations, as it is easy to make such mixtures of either fats or oils as will satisfy the ordinary requirements as to specific gravity, index of refraction, heat with sulphuric acid, iodine absorption, and saponification value.

The melting point of the fats and the fatty acids is difficult to determine, because they are mixtures of glycerids or acids, substances which have widely varying melting points. A wide difference has resulted from the varied usage of different analysts, and results obtained by exactly the same method are the only ones that are strictly comparable. For fats Wiley's method of determining melting point has been adopted by the Association of Official Agricultural Chemists.

For the free fatty acids obviously this will not do, and the capillary tube was chosen as being a method most generally used and giving the most satisfactory results.

2.—DETERMINATION OF SPECIFIC GRAVITY.^c

(a) DETERMINATION AT 15.5° C.

Determine the specific gravity of oils at 15.5° C. by the use of a pycnometer, Westphal balance,^d or accurately graduated hydrometer.^e

If determined at room temperature, the following formula may be used to calculate the specific gravity at 15.5° C.:^f

$$G = G' + .00064 (T - 15.5 \text{ C.}).$$

$$G = \text{sp. gr. at } 15.5^\circ.$$

$$G' = \text{sp. gr. at } T.$$

$$0.00064 = \text{mean correction for } 1^\circ \text{ C.}$$

^a E. E. Ewell, U. S. Dept. Agr., Div. Chem., Bul. 62, p. 125.

^b E. Spaeth, *Ztschr. anal. Chem.*, 1896, **35**, 471-493; C. A. Browne, *Jour. Am. Chem. Soc.*, 1899, **21**, 989-994.

^c See appendix, p. 149.

^d C. A. Crampton, U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 438.

^e Accurately made hydrometers reading from sp. gr. 0.900 to 0.940 at 15.5° C. will satisfy every requirement of accuracy and speed.

^f Allen, *Com. Org. Anal.*, 3d ed., vol. 2, pt. 1, p. 33; Winton, *Conn. Expt. Sta. Rept.*, pt. 2, 1900, p. 149.

This is only approximately correct, as the correction varies for different oils, but will satisfy ordinary requirements. If a higher degree of accuracy is desired, the factors given in the following table may be employed, but to obtain the best results the determination must be made at standard temperature.

Factors for calculating specific gravity.^a

Oil.	Correction for 1° C.	Observer.
Cod-liver oil.....	0.000646	A. H. Allen.
Lard oil.....	.000658	C. M. Wetherill.
Olive oil.....	.000629	C. M. Stillwell.
Arachis oil.....	.000655	A. H. Allen.
Rape oil.....	.000620	Do.
Sesame oil.....	.000624	Do.
Cotton-seed oil.....	.000629	Do.
Cocoanut olein.....	.000665	Do.

The following table gives correction for solid fats:^b

Factors for calculating specific gravity.

Fats.	Correction for 1° C.
Cocoa butter.....	0.000717
Tallow.....	.000675
Lard.....	.000650
Butter fat.....	.000617
Cocoanut stearins.....	.000674
Cocoanut oil.....	.000642
Palmit oil.....	.000657

(b) DETERMINATION AT THE TEMPERATURE OF BOILING WATER.^c

(1) *Standardization of flasks.*

First method.—Use a small specific gravity flask of from 25 to 30 cc capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper, previously heated to 100°, is then inserted, the flask removed, wiped dry, and after it has nearly cooled to room temperature placed in the balance, and weighed when balance temperature is reached.

Second method.^d—The following formula may be used for calculating the weight of water (W^T) which a given flask will hold at T° (weighed in air with brass weights at the temperature of the room) from the weight of water (W^t) (weighed in air with brass weights at the temperature of the room) contained therein at t° :

$$W^T = W^t \frac{d^T}{d^t} [1 + \gamma (T - t)]$$

d^T =the density of water at T° .

d^t =the density of water at t° .

γ =the coefficient of cubical expansion of glass.^e

^a Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 33.

^b Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 32.

^c U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 33.

^d E. E. Ewell, U. S. Dept. Agr., Div. Chem., Bul. 62, p. 125.

^e This factor is commonly given as 0.000026, but it varies considerably. Schulze (Ztschr. anal. Chem., 1882, 21, 167-177) found the glass used by him varied from 0.0000288 to 0.0000305; an average of these is 0.0000296. Ewell has used 0.000028 in his work, U. S. Dept. of Agr., Div. of Chem., Bul. 62, p. 121.

(2) *Determination.*

Weight of fat at the temperature of boiling water.—The flask is rinsed with alcohol and ether, and dried for a few minutes at the temperature of boiling water. It is filled with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles, replaced in the water bath, and kept for thirty minutes at the temperature of boiling water. The stopper, previously heated to 100° C, is inserted, the flask removed, wiped dry, placed in the balance after it has nearly cooled to room temperature, and weighed when the balance temperature is reached. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example:

	Grams.
Weight of flask, dry	10.0197
Weight of flask, plus water	37.3412
Weight of water	27.3215
Weight of flask, plus fat	34.6111
Weight of fat	24.5914
Specific gravity = $24.5914 \div 27.3215 = 0.90008$.	

The weight of the flask dry and empty may be used constantly if great care be taken in handling and cleaning the apparatus, but the weight of water at boiling temperature must be determined under the barometric conditions prevailing at the time the determination is made.

Example:

	Grams.
Weight of flask, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

3.—DETERMINATION OF INDEX OF REFRACTION.^a

Determine the index of refraction with any standard instrument, oils being read at 15.5° C. and fats at 40° C.

The temperature must be controlled with great care, and in accurate work the readings should be taken at standard temperature. The readings of the Zeiss butyro-refractometer can be reduced to standard temperature by following formula:^b

$$R = R' + .55 (T' - T).$$

in which R is the reading reduced to T, R' the reading at Temp. T, T the standard temperature, and .55 the correction for 1° C. in scale divisions. With oils the factor .58 is substituted in the formula for .55, since they have a higher index of refraction.

To calculate to standard temperature the readings of the instruments which give index of refraction directly the factor 0.000365 may be used. As the temperature rises the refractive index falls. Example: The refractive index of a butter fat determined at 32.4° C. = 1.4540 is reduced to 25° C., as follows: $32.4 - 25 = 7.4$; $0.000365 \times 7.4 = 0.0027$; it is then $1.4540 + 0.0027 = 1.4567$.

The instrument used should be set with distilled water at 18°C., the theoretical refractive index of water at that temperature being 1.3330. In the determination above given the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given 1.4597.

The index of refraction varies greatly with the specific gravity, increasing as it increases. In abnormal results it is often well to see if the specific refractive power^c

^a See appendix, p. 150.

^b Wiley, Prin. and Prac. of Agri. Anal., vol. 3, p. 341. Winton, Conn. Expt. Sta. Rept., 1900, pt. 2, p. 142.

^c Landolt, Ber., 1882, 15, 1031. C. A. Browne, Jour. Am. Chem. Soc., 1899, 21, 991.

is different from the normal. Calculate the specific refractive power from the formula $\frac{N-1}{D}$,^a in which N equals the refractive index and D the specific gravity. Always state temperature at which the determinations were made.

(a) ABBE'S REFRACTOMETER.

A later and much improved model of the Abbe instrument, in which arrangements are made for controlling the temperature, the weakness of the older form,^b is described in Benedikt.^c

(b) ZEISS BUTYRO-REFRACTOMETER.^d

Place the instrument (fig. 1) upon a table where diffuse daylight or any form of artificial light can be readily admitted for illumination. Supply through nozzle D a

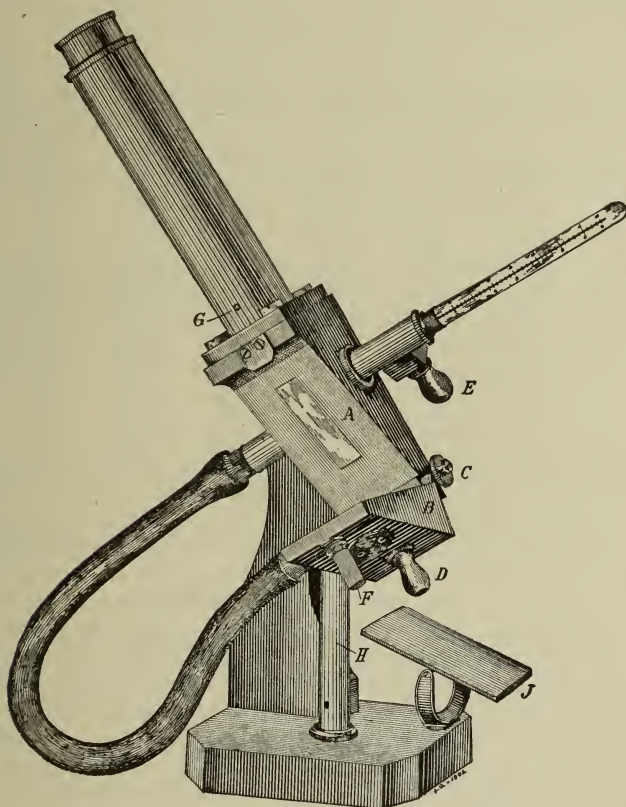


FIG. 1.—Zeiss's butyro-refractometer.

stream of water of constant temperature. Then open the prism casing by giving to the pin F a half turn. The surfaces of the prism must now be cleaned with the greatest care, which is best done by applying soft linen moistened with ether. Now

^a H. R. Procter, Jour. Soc. Chem., Ind., 1898, 17, 1021-1026, has shown that the Lorenz formula $\frac{N^2-1}{(N^2+2)D}$ gives much more satisfactory results than $\frac{N-1}{D}$, and gives table for calculation.

^b For a description of the older form of the Abbe instrument, see U. S. Dept. Agr., Div. Chem., Bul. 46, revised, p. 49.

^c Anal. der. Fette u. Wach., 3d ed., p. 105.

^d Wiley, Prin. and Prac. Agr. Anal., vol. 3, pp. 339-341. Also description by manufacturer.

melt the sample of fat and pour the clear fat through a filter, allowing the first two or three drops to fall on the surface of the prism contained in casing B (oils must be filtered if turbid). For this purpose the apparatus should be raised with the left hand, so as to place the prism surface in a horizontal position. Then press B against A and bring F back into its original position by turning it in the opposite direction. Adjust the mirror until it gives the sharpest reading. If the reading be not distinct after running water of a constant temperature through the instrument for some time, the fat is not evenly distributed on the surfaces of the prism and the process must be repeated. The instrument should be carefully adjusted by means of the standard fluid which is supplied. As the index of refraction is greatly affected by temperature, care must be used to keep it constant.

The following table can be used to convert the degrees of the instrument into refractive indices:

Butyro-refractometer readings and indices of refraction. ^a

Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.	Reading.	Index of refraction.
40.0	1,4524	50.0	1,4593	60.0	1,4659	70.0	1,4723
40.5	1,4527	50.5	1,4596	60.5	1,4662	70.5	1,4726
41.0	1,4531	51.0	1,4600	61.0	1,4665	71.0	1,4729
41.5	1,4534	51.5	1,4603	61.5	1,4668	71.5	1,4732
42.0	1,4538	52.0	1,4607	62.0	1,4672	72.0	1,4735
42.5	1,4541	52.5	1,4610	62.5	1,4675	72.5	1,4738
43.0	1,4545	53.0	1,4613	63.0	1,4678	73.0	1,4741
43.5	1,4548	53.5	1,4616	63.5	1,4681	73.5	1,4744
44.0	1,4552	54.0	1,4619	64.0	1,4685	74.0	1,4747
44.5	1,4555	54.5	1,4623	64.5	1,4688	74.5	1,4750
45.0	1,4558	55.0	1,4626	65.0	1,4691	75.0	1,4753
45.5	1,4562	55.5	1,4629	65.5	1,4694	75.5	1,4756
46.0	1,4565	56.0	1,4633	66.0	1,4697	76.0	1,4759
46.5	1,4569	56.5	1,4636	66.5	1,4700	76.5	1,4762
47.0	1,4572	57.0	1,4639	67.0	1,4704	77.0	1,4765
47.5	1,4576	57.5	1,4642	67.5	1,4707	77.5	1,4468
48.0	1,4579	58.0	1,4646	68.0	1,4710	78.0	1,4771
48.5	1,4583	58.5	1,4649	68.5	1,4713	78.5	1,4774
49.0	1,4586	59.0	1,4652	69.0	1,4717	79.0	1,4777
49.5	1,4590	59.5	1,4656	69.5	1,4720	79.5	1,4780

^b Winton, Conn. Expt. Sta., Rept., 1900, pt. 2, p. 143.

4.—DETERMINATION OF IODIN ABSORPTION, HÜBL'S METHOD. ^a

(a) PREPARATION OF REAGENTS.

Iodin solution.—Dissolve 25 grams of pure iodine in 500 cc of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc of 95 per cent alcohol. The latter solution, if necessary, is filtered, and then the two solutions are mixed. The mixed solution should be allowed to stand twelve hours before using.

Decinormal sodium thiosulfate solution.—Dissolve 24.8 grams of chemically pure sodium thiosulfate freshly pulverized as finely as possible and dried between filter or blotting paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made.

Starch paste.—One gram of starch is boiled in 200 cc of distilled water for ten minutes and cooled to room temperature.

Solution of potassium iodid.—One hundred and fifty grams of potassium iodid are dissolved in water and made up to 1 liter.

Solution of potassium bichromate.—Dissolve 3.874 grams of chemically pure potassium bichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made. The bichromate solution should be checked against pure iron.

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 50.

(b) DETERMINATION.

(1) *Standardizing the sodium thiosulfate solution.*

Place 20 cc of the potassium bichromate solution, to which has been added 10 cc of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cc of strong hydrochloric acid. / Allow the solution of sodium thiosulfate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium thiosulfate solution until the blue color just disappears. The number of cubic centimeters of thiosulfate solution used multiplied by 5 is equivalent to 1 gram of iodine.

Example: Twenty cubic centimeters of bichromate solution required 16.2 cc sodium thiosulfate; then $16.2 \times 5 = 81 = \text{number cubic centimeters of thiosulfate solution equivalent to 1 gram of iodine}$. Then 1 cc thiosulfate solution = 0.0127 gram of iodine. Theory for decinormal solution of sodium thiosulfate 1 cc = 0.0127 gram of iodine.

(2) *Weighing the sample.^a*

Weigh about 1 gram of fat or 0.500 gram of oil^b on a small watch crystal^c or by other suitable means. The fat is first melted, mixed thoroughly, poured onto the crystal and allowed to cool.

Introduce the watch crystal into a wide-mouth 16-ounce bottle with ground-glass stopper.

(3) *Absorption of iodine.*

The fat or oil in the bottle is dissolved in 10 cc of chloroform. After complete solution has taken place, 30 cc of the iodine solution are added in the case of fats, or from 40 to 50 cc^d in the case of oils. Place the bottle in a dark place and allow to stand, with occasional shaking, for three hours.^e This time must be closely adhered to in order to get good results. The excess of iodine should be at least as much as is absorbed.

(4) *Titration of the unabsorbed iodine.*

Add 20 cc of the potassium iodid solution, and then 100 cc of distilled water to the contents of the bottle. Wash any iodine which may be noticed upon the stopper back into the bottle with the potassium iodid solution. Titrate the excess of iodine with the sodium thiosulfate solution, which is added gradually, with constant shaking, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste, and continue the titration until the blue color has entirely disappeared. Toward the end of the reaction stopper the bottle and shake violently, so that any iodine remaining in solution in the chloroform may be taken up by the potassium iodid solution. The excess of sodium thiosulfate solution should be sufficient to prevent a reappearance of any blue color in the flask for five minutes.

(5) *Setting the value of iodine solution by thiosulfate solution.*

At the time of adding the iodine solution to the fat, two bottles of the same size as those used for the determination should be employed for conducting the operation described above, but without the presence of any fat. In every other respect the

^a The writer has found it unsatisfactory to weigh so small amounts of fat in flask as directed in the A. O. A. C. methods.

^b With drying oils which have a very high absorbent power, 0.100 to 0.200 gram should be taken.

^c See appendix, p. 150.

^d F. Ulzer, Jour. Soc. Chem. Ind., 1898, 17, 276, says iodine should be in excess about twice the amount that is absorbed. The solution loses strength with age, but can be used so long as 35 cc of decinormal thiosulfate neutralize 25 cc iodine solution.

^e The time allowed does not give the complete iodine absorption power of an oil or fat and can not be compared with determinations where six to twelve hours have been used. It gives very satisfactory comparative results, but the time factor must be very closely adhered to.

performance of the blank experiments should be just as described. These blank experiments must be made each time the iodine solution is used.

Example blank determinations: Forty cc iodine solution required 62.05 cc of sodium thiosulphate solution. Forty cc iodine solution required 62.15 cc of sodium thiosulphate solution. Mean, 62.1 cc.

Per cent of iodine absorbed:

Weight of fat taken.....	grams..	1.0479
Quantity of iodine solution used	cubic centimeters..	40.0
Thiosulfate equivalent to iodine used	do....	62.1
Thiosulfate equivalent to remaining iodine.....	do....	30.2
Thiosulfate equivalent to iodine absorbed.....	do....	31.9

Per cent of iodine absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

5.—DETERMINATION OF SAPONIFICATION NUMBER AND SOLUBLE AND INSOLUBLE ACIDS.^a

The saponification number, and soluble and insoluble acids, are determined in one sample by the following method:

(a) PREPARATION OF REAGENTS.^b

Standard sodium hydroxid solution.—A decinormal solution of sodium hydroxid is used. Each cubic centimeter contains 0.0040 gram of sodium hydroxid and neutralizes 0.0088 gram of butyric acid ($C_4H_8O_2$).

Alcoholic potash solution.—Dissolve 40 grams of good potassium hydroxid in 1 liter of 95 per cent redistilled alcohol.^c The solution must be clear and the potassium hydroxid free from carbonates.

Standard acid solution.—Prepare accurately a half normal solution of hydrochloric acid.

Indicator.—Dissolve 1 gram of phenolphthalein in 100 cc of 95 per cent alcohol.

(b) WEIGHING OF SAMPLE.

The saponification is carried on in a wide-mouth Erlenmeyer flask holding from 250 to 300 cc. These are cleaned by thoroughly washing with water, alcohol, and ether, wiped perfectly dry on the outside, and heated for one hour at the temperature of boiling water. The flasks are then placed on a tray, covered with a silk handkerchief, and allowed to cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed.

About 5 grams of the melted fat, which has been filtered, is run in by means of a pipette, and after cooling the flask and contents are again weighed.^d

(c) KOETSTORFER OR SAPONIFICATION NUMBER.^e

Measure 50 cc of the alcoholic potash solution into the flask by means of a burette or pipette, which is allowed to drain a definite time. Connect the flask with a reflux^f condenser and boil for thirty minutes, when the fat is completely saponified. Cool the flask and titrate with half-normal hydrochloric acid, using phenolphthalein as indicator. The Koetstorfer number (milligrams of potassium hydroxid required to saponify 1 gram of fat) is obtained by subtracting the number of cubic centimeters of hydrochloric acid used to neutralize the excess of alkali after saponification

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 47.

^b See appendix, p. 150.

^c The alcohol should be redistilled from potassium hydroxid on which it has been standing for some time, or with which it has been boiled for some time, using a reflux condenser.

^d See appendix, p. 150.

^e Chiefly of value in oil work in the detection of rape-oil, resin, and paraffin products.

^f Almost any sort of a reflux condenser will do. A small funnel placed in the mouth of the flask is perfectly satisfactory and very convenient.

from number of cubic centimeters necessary to neutralize the 50 cc of alkali added, multiplying the result by 28.06 (mg. potassium hydroxid per cubic centimeter) and dividing by the number of grams of fat used.

To calculate the saponification equivalent^a divide 56,100 by the saponification number, the saponification equivalent being the number of grams of fat saponified by one equivalent of potassium hydroxid, or 56.1 grams. There is no advantage in stating it in this way, and for sake of uniformity, the Koetstorfer number being more generally used, it would seem advisable to adopt it.

(d) SOLUBLE ACIDS.

Place the flask on a water bath and evaporate the alcohol. Add such an amount of half-normal hydrochloric acid that its volume plus the amount used in titrating for the saponification number will be 1 cubic centimeter in excess of the amount required to neutralize the 50 cc of alcoholic potash added. Connect the flask with a condensing tube 3 feet long made of small glass tubing and place it on the steam bath until the separated fatty acids form a clear stratum on the upper surface of the liquid. Fill the flask to the neck with hot water and cool it in ice water until the cake of fatty acids is thoroughly hardened. Pour the liquid contents of the flask through a dry weighed filter into a liter flask, taking care not to break the cake. Fill the flask again with hot water, set on steam bath until the fatty acids collect at the surface, cool by immersing in ice water, and filter the liquid again into the liter flask. Repeat this treatment with hot water, followed by cooling and filtration of the wash water three times, collecting the washings in the liter flask, and titrate with deci-normal alkali, using phenolphthalein as indicator.

The number of cubic centimeters of deci-normal alkali used in this titration diminished by 5 (corresponding to the excess of 1 cc of half-normal acid) and multiplied by 0.0088 gives the weight of butyric acid in the amount of fat saponified; dividing this by the weight of fat taken gives the percentage of soluble acids.

(e) INSOLUBLE ACIDS OR HEHNER NUMBER.

Allow the flask containing the cake of insoluble acids and the filter paper through which the soluble acids have been filtered to drain and dry for twelve hours in the air. Transfer the filter paper to the flask and dry the flask and contents for three hours in a water-jacketed oven, cool, and weigh. Then dry for another two hours, cool, and weigh. If there be any considerable decrease in weight, repeat the drying. The weight obtained less the weight of the filter paper gives weight of insoluble acids, from which the percentage can be easily calculated.

6.—DETERMINATION OF FREE FATTY ACIDS.^b

Weigh 20 grams of fat or oil into a flask, add 50 cc of 95 per cent alcohol which has been neutralized with weak caustic soda, using phenolphthalein as indicator, and heat to boiling point. Agitate the flask thoroughly in order to dissolve the free fatty acids as completely as possible. Titrate with deci-normal alkali, agitating thoroughly until the pink color persists after vigorous shaking.

Express results either as percentage of oleic acid, as acid degree (cubic centimeters of normal alkali required to neutralize the free acids in 100 grams of oil or fat), or as acid value (milligrams of potassium hydroxid required to saturate the free acids in 1 gram of fat or oil).

1 cc deci-normal alkali=0.0282 grams oleic acid.

7.—DETERMINATION OF VOLATILE ACIDS OR REICHERT-MEISSL NUMBER.

See methods for dairy products p. 38.

^a Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, pp. 53-55.

^b Allen, Com. Org. Anal., 3d ed., vol. 2, p. 105.

8.—FOR ESTIMATION OF LIQUID AND SOLID FATTY ACIDS, MUTER'S METHOD^a MODIFIED BY LANE.^b

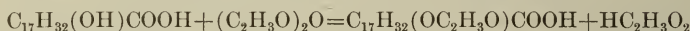
Weigh 5 grams of oil or fat into an Erlenmeyer flask, saponify, precipitate with lead acetate, and extract with ether, as directed under determination of arachidic acid. Filter the ether solution of soluble lead soap into a Muter tube or separatory funnel and decompose the soap by shaking with 40 cc of a 1:5 solution of hydrochloric acid. The soap is completely decomposed when the ether becomes clear and colorless.

The lead chlorid is drawn off from the ether solution and the ether washed free from acid. An aliquot of this ether solution is evaporated free from ether in an atmosphere of carbon dioxid in order to prevent the oxidation of the oleic acid, and weighed to determine the per cent of liquid acids; .2 to .3 gram of this is weighed and the iodine number determined in the ordinary way.

As it is very difficult to dry the oleic acid without very serious oxidation, it is just as satisfactory to determine the weight of insoluble acids by the following method: Wash the insoluble soap left on the filter into a flask, decompose with hydrochloric acid, and heat until the fatty acids are melted. Fill the flask with hot water, cool, pour off the water, and wash again the solidified fatty acids. Dissolve them in hot 95 per cent alcohol, transfer to weighed dish, remove the alcohol by evaporation, dry, weigh, and calculate the percentage of solid fatty acids.

9.—DETERMINATION OF ACETYL VALUE.^c

Benedikt proposed to determine the hydroxy acids and alcohols by the use of acetic anhydrid $(C_2H_3O)_2O$ as illustrated in the following reaction:^d



He proposed to work on the fatty acids, but the process was modified by Lewkowitsch^e who works on the oils or fats directly, which gives more exactly the true content of hydroxy acids.^f

The procedure is as follows:

Boil the oil or fat with an equal volume of acetic anhydrid $(C_2H_3O)_2O$ for two hours and pour the mixture into a large beaker containing 500 cc of water and boil for half an hour. To prevent bumping, a slow current of carbonic acid is passed into the liquid through a finely drawn out tube reaching nearly to the bottom. Allow the mixture to separate into two layers, siphon off the water, and boil the oily layer with fresh water until it is no longer acid to litmus paper.

The acetylated fat is then separated from the water and dried and filtered in a drying oven.

Weigh from 2 to 4 grams of the acetylated fats into a flask and saponify with alcoholic potash as in the determination of saponification equivalent. If the distillation process is to be adopted it is not necessary to work with a standardized alcoholic potash solution. In case the filtration method is used, which will be found much shorter, it is necessary that the alcoholic potash should be measured exactly.

In either case evaporate the alcohol after saponification and dissolve the soap in water. Now two procedures are possible—either distillation or filtration.

(a) DISTILLATION PROCESS.

Acidify with dilute sulphuric acid (1-10) and distill the liquid as in the Reichert test. As several hundred cubic centimeters must be distilled, either a current of

^a J. Muter and L. L. De Koningh, *Analyst*, 1889, **14**, 61.

^b N. J. Lane, *Jour. Am. Chem. Soc.*, 1893, **15**, 110.

^c Lewkowitsch, *Jour. Soc. Chem. Ind.*, 1897, **16**, 503-506; Benedikt, *Analyse der Fette u. Wach.*, 3d ed., p. 146; Allen, *Com. Org. Anal.*, 3d ed., 2, pt. 1, pp. 66-67.

^d Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 127.

^e *Jour. Soc. Chem. Ind.*, 1897, **16**, 503.

^f J. Lewkowitsch, *Jour. Soc. Chem. Ind.*, 1890, **9**, 846.

steam is run through or portions of water are added from time to time. From 500 to 700 cc of distillate will be found to be sufficient. Filter the distillates to remove any insoluble acids carried over by the steam, and titrate the filtrate with deci-normal potassium hydroxid, using phenolphthalein as indicator. Multiply the number of cubic centimeters of alkali employed by 5.61 and divide by the weight of substance taken. This gives the acetyl value.

(b) FILTRATION PROCESS.

Add to the soap solution a quantity of standard sulphuric acid exactly corresponding to the amount of alcoholic potash added, warm gently, and the free fatty acids will collect on top.

Filter off the liberated fatty acids, wash with boiling water until the washings are no longer acid, and titrate the filtrate with deci-normal potassium hydroxid, using phenolphthalein as indicator. Calculate the acetyl value as before.

10.—DETERMINATION OF PHYTOSTEROL AND CHOLESTEROL.^a

Boil 50 grams of fat or oil in a flask with reflux condenser with 75 cc of 95 per cent alcohol for five minutes and separate alcoholic solution. Repeat with another portion of alcohol and separate. Mix the alcoholic solution with 15 cc of 30 per cent sodium hydroxid and boil in a flask with a condensation tube until one-fourth of the alcohol is evaporated. Evaporate nearly to dryness in porcelain dish and shake the residue with ether. The ethereal solution is evaporated to dryness, taken up with a little ether, filtered, again evaporated, dissolved in hot 95 per cent alcohol and allowed to crystallize.

Cholesterol can easily be distinguished from phytosterol by the form and grouping of the crystals; also by the melting point, which is 146° C.,^b while that of phytosterol is from 130° to 137.5° C.^c

Phytosterol is found in most vegetable oils, with the notable exception of olive and palm oil. The crystals as separated from hot alcohol appear in tufts of needles.

Cholesterol is characteristic of animal fats. It crystallizes in thin rhombic tables.

11.—DETERMINATION OF THE UNSAPONIFIABLE RESIDUE.^d

Saponify 5 grams^e of oil or fat with alcoholic potassium hydroxid and remove the alcohol by evaporation. Wash into separatory funnel with from 70 to 100 cc of water and extract with from 50 to 60 cc of ether. In case the two liquids do not separate, a few cubic centimeters of alcohol may be added. Separate the water solution and wash the ether with water containing a few drops of sodium hydroxid. Again extract the soap solution and washings with ether and evaporate the combined extracts to dryness. In most cases it is advisable to add a little alcoholic potassium hydroxid to the residue and heat in order to saponify any traces of fats left unsaponified and extract again with ether. Transfer to a weighed dish and dry as quickly as possible in a water oven.

Many of the hydrocarbon oils are volatile at 100° C., so that the drying should not be carried any further than necessary. With resin oil, paraffin wax, and the denser mineral oils there is little danger of loss at 100°.

On account of the solubility of soap in ether and petroleum ether it is well to wash the residue with warm water containing a little phenolphthalein. If it shows alkaline reaction there is soap present.

^a Forster and Reichelmann *Analyst*, 1897, **22**, 131; E. Salkowski, *Ztsch. anal. Chem.*, 1887, **26**, 557; E. Von Raumer, *Ztsch. angew. Chem.*, 1898, **13**, 555-556; *Jour. Soc. Chem. Ind.*, 1898, **17**, 774; H. Kreis and O. Wolf, *Jour. Soc. Chem. Ind.*, 1898, **17**, 1075.

^b E. Salkowski, *Ztschr. anal. Chem.*, 1887, **26**, 557.

^c Bömer, *Ztschr. Unter. d. Nahr u. Genuss*, 1898, **1**, 81.

^d Allen, *Com. Org. Anal.*, 3d Ed., Vol. **2**, pp. 1 and 113.

^e See Appendix, p. 150.

12.—DETERMINATION OF MELTING POINTS OF FATS^a—WILEY'S METHOD.^b

(a) PREPARATION OF REAGENTS.

Have a piece of ice floating in distilled water that has been recently boiled. Prepare a mixture of alcohol and water of the same specific gravity as the fat to be examined. This is done by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot, the water is poured into the test tube described below (2) until it is nearly half full. The test tube is nearly filled with the hot alcohol, which is carefully poured

down the side of the inclined tube to avoid too much mixing. If the alcohol is not added until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

(b) APPARATUS.

The apparatus for determining the melting point consists of an accurate thermometer reading easily tenths of a degree; a cathetometer for reading the thermometer (but this may be done with an eyeglass if held steadily and properly adjusted); an ordinary thermometer; a tall beaker 35 cm high and 10 cm in diameter; a test tube 30 cm long and 3.5 cm in diameter; a stand for supporting the apparatus; some method of stirring the water in the beaker (for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker). (See fig. 2.)

(c) DETERMINATION.

The disks of fat are prepared as follows: The melted and filtered fat is allowed to fall from

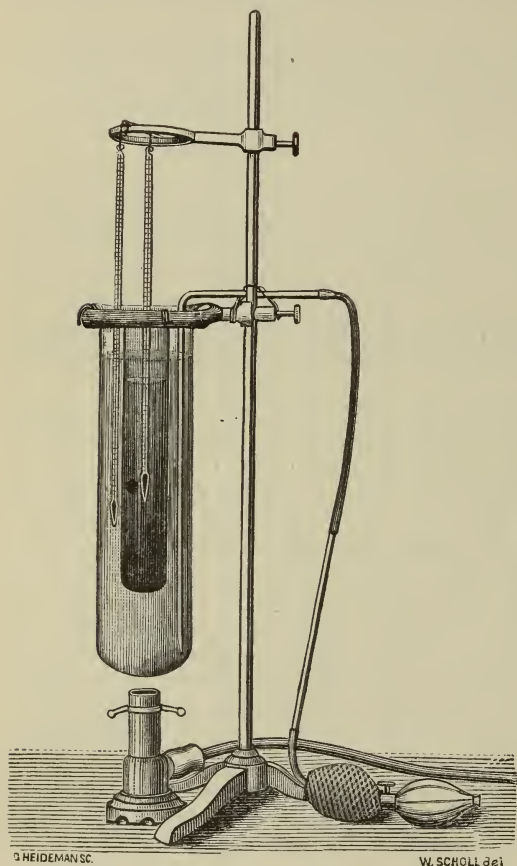


FIG. 2.—Apparatus for the determination of the melting point.

a dropping tube from a height of from 15 to 20 cm on a smooth piece of ice floating in distilled water that has been recently boiled. The disks thus formed are from 1 to 1.5 cm in diameter, and weigh about 200 mg. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The disks must be allowed to stand for two or three hours in order to obtain the normal melting point.

The test tube containing the alcohol and water is placed in a tall beaker contain-

^a See Appendix, p. 151.

^b U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 52.

ing water and ice, until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° C. below the melting point, the disk of fat begins to shrivel and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2° C. of the increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and, when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken the tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The edge of the disk should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place and the disk adhere to the sides of the tube a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example: Melting point of sample of butter:	Degrees.
First trial	33. 15 C.
Second trial	33. 05 C.
Third trial	33. 10 C.

13.—DETERMINATION OF MELTING POINT OF FATTY ACIDS.^a

Draw up the melted fatty acid into a very thin-walled capillary tube 1 or 2 inches long according to the length of bulb of the thermometer used. Seal one end of the tube and allow the fatty acid to cool on ice for from twelve to fifteen hours. Then attach to the bulb of a delicate thermometer graduated to one-fifth degree, immerse in a beaker of water, and warm up very slowly. The point where the acid becomes transparent is taken as the melting point.

14.—DETERMINATION OF MAUMENÉ NUMBER.^b

The following apparatus has been largely used by the writer and has given very satisfactory results:

A beaker, 5 inches by $1\frac{1}{2}$ inches, is placed inside of another 6 inches by 3 inches, and a wet mixture of asbestos and plaster of paris tightly packed around the inner beaker. This, when dried, makes a hard, solid packing which radiates heat very slowly.

^aU. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 448. Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 97. Wiley, Prin. and Prac. Agr. Anal., vol. 3, p. 321.

^bAllen, *Com. Org. Anal.*, 3d ed., vol. 2, pt. 1, p. 76.

Remove the inner beaker, weigh into it 50 grams of fat, and note the temperature carefully. Then, from a pipette which will deliver it in approximately one minute, add 10 cc of the strongest sulphuric acid^a which is at the same temperature as the oil.

While the acid is being introduced, stir the oil and acid with an accurate thermometer. Then hold the thermometer bulb carefully in the center of the mixture, and when the mercury reaches the highest point note the reading. It is easy to determine this point as the column of mercury remains stationary for some time. It is necessary to take care not to read the temperature too soon, as some oils take considerable time to reach their maximum point.

The difference between the initial reading and the final reading, expressed in degrees centigrade, gives the Maumené number.

Great care must be taken to have the acid of the highest strength. With the semi-drying oils, such as cotton-seed, the use of this strength of acid will cause foaming and make it almost impossible to obtain the correct rise of temperature. With such oils, either a weaker acid will have to be used and the results compared with the rise of temperature with water, or a dilution with paraffin oil made. It is always best to test the apparatus and acid by use of water and oils of known purity. With 50 grams of water and 10 cc of 99 per cent sulphuric acid, Thomson and Ballantyne^b obtained a rise of 46.5° C. Working with acid of specific gravity of 1.844, the average of a number of determinations in this laboratory was 45°, but this will vary with the apparatus and manipulator.

The acid which is used in testing the apparatus should be used in all the determinations and care should be taken that it does not lose its strength. When this test is conducted with care, it is one of the most valuable in detection of adulteration in fats and oils.

In reporting results obtained, the rise of temperature with water should be stated, otherwise no comparative value can be attached to the results.

15.—DETERMINATION OF RESIN OIL.

Take the pure oil or a definite dilution with petroleum ether and polarize in a 200 mm tube.

Resin oil has a polarization of from +30 to +40 on the sugar scale (Schmidt and Haensch) in a 200 mm tube while other oils read between 1°+ and -1°.

16.—HALPHEN^d REACTION FOR COTTON-SEED OIL.

Carbon disulphid, containing about 1 per cent of sulphur in solution, is mixed with an equal volume of amyl alcohol. Mix equal volumes of this reagent and the oil under examination and heat in a bath of boiling brine for fifteen minutes. In the presence of as little as 1 per cent of cotton-seed oil, an orange or red color is produced, which is characteristic.

Lard and lard oil from animals fed on cotton-seed meal will give a faint reaction; also the fatty acids.

This test is more sensitive than the Bechi test and less liable to give unsatisfactory results in the hands of an inexperienced person. It is not affected by rancidity. The depth of color is proportional, to a certain extent, to the amount of oil present, and by making comparative tests with cotton-seed oil some idea as to the amount present can be obtained, but it must be remembered that different oils react with different intensities, and oils which have been heated to 200° to 210° C.^d react with greatly

^aSee appendix, p. 151.

^bJour. Soc. Chem. Ind., 1891, 10, 234.

^cG. Halphen, Jour. Pharm. Chim., 1897, 6, 390-391. Analyst, 1897, 22, 326. Allen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 143. Winton, Conn., Exp. Sta. Rept., 1900, pt. 2, p. 144.

^dAllen, Com. Org. Anal., 3d ed., vol. 2, pt. 1, p. 143.

diminished intensity. Heating ten minutes at 250° renders cotton-seed oil incapable of giving reaction.^a

17.—BECCHI OR SILVER NITRATE TEST FOR COTTON-SEED OIL.^b

Reagent.^c—Dissolve 2 grams of silver nitrate in 200 cc of 95 per cent alcohol and 40 cc of ether, adding 1 drop of nitric acid.

Mix 10 cc of oil or melted fat, 5 cc of reagent, and 10 cc of amyl alcohol^d in a test tube. Divide, heat one-half in a boiling water bath for ten minutes, and then compare with portion not heated. Any blackening due to reduced silver shows presence of cotton-seed oil.

Other oils which have become rancid,^e and lards which have been steamed or heated at high temperature, contain decomposition products which have a reducing action on silver nitrate. The writer found in testing a large number of salad oils some which contained no cotton-seed oil, according to the Halphen test, but gave a brown coloration with Bechi reagent, and in some cases reduced silver. These same oils on being purified gave no reaction. Hence the oils or fats should be purified before testing.

To purify the oils and fats, heat from 20 to 30 grams on water bath for a few minutes with the addition of 25 cc of 95 per cent alcohol,^f shake thoroughly, decant as much of the alcohol as possible, and wash with 2 per cent nitric acid,^g and finally with water. The oil or lard thus purified will give no reduction at all if it contains no cotton-seed oil. Heating the oils or fats to 100° C. or simple washing with 2 per cent nitric acid is not sufficient except in a few cases.

With oils the use of the Halphen and Bechi tests will be found to be useful as a means of approximately determining the amounts of adulteration present. If Halphen gives a reaction and Bechi does not, the adulteration with cotton-seed oil is probably less than 20 per cent.

18.—RENARD'S^h TEST FOR PEANUT OIL AS MODIFIED BY TOLMAN.

Weigh 5 grams of oil into an Erlenmeyer flask, as directed under determination of saponification number. (If this is accurately weighed and a standard solution of alcoholic potash used, the saponification number can be determined on the same sample by titrating the excess of alcoholic potash used in the saponification with half-normal acetic acid.) Saponify with alcoholic potash, naturalize exactly with dilute acetic acid, using phenolphthalein as indicator, and wash into a 500-cc flask containing a boiling mixture of 200 cc of water, and 60 cc of a 10 per cent lead acetate solution. Boil for a minute, and then cool the precipitated soap by immersing the flask in water, occasionally giving the flask a whirling motion to cause the soap to stick to the sides of the flask. After the soap has cooled, the water and excess of lead can be poured off, and the soap washed with cold water and with 90 per cent (by volume) alcohol.ⁱ Now, add 200 cc of ether, cork the flask, and allow to stand for some time until the soap is disintegrated, then heat on the water bath, using a reflux condenser, and boil for about five minutes. In the oils most of the soap will

^a D. Holde and R. Pelgry, *Jour. Soc. Chem. Ind.*, 1899, 18, 711.

^b See appendix, p. 151.

^c Pearmain and Moor, *Allen Com. Org. Anal.*, 3 ed., vol. 2, pt. 1, p. 143. Wesson, *Jour. Am. Chem. Soc.*, 1895, 17, 724.

^d The addition of amyl alcohol is not necessary, but the writer finds it very convenient, as it dissolves the oils or fats and enables one to mix the oil and reagent much better.

^e Wesson, *Jour. Am. Chem. Soc.*, 1895, 17, 724. A. L. Winton, *Conn. Expt. Sta. Rept.*, 1900, pt. 2, p. 143.

^f Used by the writer and found to be much more convenient and just as satisfactory as dilute alkali.

^g Wesson, *Jour. Am. Chem. Soc.*, 1895, 17, 724.

^h Renard, *Comp. Rend.*, 1871, 73, 1330. Benedikt and Lewkowsitch, *Oils, Fats, and Waxes*, p. 365.

ⁱ Process used by N. J. Lane in his modification of Muter's method. *Jour. Am. Chem. Soc.*, 1893, 15, 110.

be dissolved, while in lards, where there is so much stearin, part will be left undissolved. Cool the ether solution of soap down to from 15° to 17° C., and allow to stand until all the insoluble soaps have crystallized out. It should stand about twelve hours.

Now, filter and wash the precipitate with ether. Save the filtrate for the determination of the iodine number of the liquid fatty acids by the Muter method.

The soaps on the filter are washed back into the flask by means of a stream of hot water acidified with hydrochloric acid.

Add an excess of dilute hydrochloric acid, fill up the flask with hot water, allow the free fatty acids to harden and separate from the precipitated lead chlorid, wash, drain, and dissolve the fatty acids in 25 cc of boiling 90 per cent (by volume) alcohol. The crystals of arachidic acid separate out as the liquid cools. From 5 to 10 per cent of peanut oil can be detected by this method, as it effects a complete separation of the soluble acids from the insoluble, which interfere with the crystallization of the arachidic acid. Filter, wash the precipitate twice with 10 cc of 90 per cent (by volume) alcohol, and then with alcohol of 70 per cent (by volume). Dissolve off the filter with boiling absolute alcohol, evaporate to dryness in a weighed dish, dry and weigh. Add to this weight 0.0025 gram for each 10 cc of 90 per cent alcohol used in the crystallization and washing if done at 15° C., if done at 20°, 0.0045 gram for each 10 cc.

The melting point of arachidic acid obtained in this way is between 71° and 72° C. Twenty times the weight of arachidic acid will give the approximate amount of peanut oil present.

Another method^a which gives as satisfactory an approximation of the amount of peanut oil present is to allow the arachidic^b acid to crystallize in a 100 cc graduated cylinder and measuring the volume of the precipitate. This volume will have to be determined for the working temperature and the length of the time by use of known mixtures of peanut oil. Cotton-seed and lard oil give slight precipitates when treated by this method.

Arachidic acid has a characteristic structure and can be detected by the microscope.

No examination of olive oil is complete without making the test for peanut oil, which is probably a common adulterant, especially in French and Italian oils.

19.—BAUDOUIN TEST FOR SESAME OIL.

Dissolve 0.1 gram of finely powdered sugar in 10 cc of hydrochloric acid (sp. gr. 1.20), add 20 cc of the oil to be tested, shake thoroughly for a minute and allow to stand. The aqueous solution separates almost at once. In the presence of even a very small admixture of sesame oil, this is colored crimson. Some olive oils give a slight pink coloration with this reagent, but they are not hard to distinguish if comparative tests with sesame oil are made.

20.—VILLIVECCHIA^c TEST FOR SESAME OIL.

Mix 2 grams of furfural with 100 cc alcohol (95 per cent), and take 0.1 cc of this solution, 10 cc hydrochloric acid (sp. gr. 1.20), and 10 cc of oil and mix thoroughly by shaking in a test tube and the same color is developed as when the sugar is used. Villivecchia attributed the Baudouin test to the formation of furfural from the action of levulose and hydrochloric acid, and so substituted furfural for sucrose.

As furfural and hydrochloric acid give a violet tint with hydrochloric acid, it is necessary to use the very dilute solution given in the method.

^a Suggested by W. D. Bigelow.

^b As the solubility of arachidic acid in 90 per cent alcohol increases very rapidly with the temperature, care must be taken to keep the temperature of crystallization down to between 15° and 20° C., and to obtain satisfactory results the temperature must be same as used in the standards.

^c Villivecchia and Fabris, *Journ. Soc. Chem. Ind.*, 1893, **12**, 97 and 1894, **13**, 69. Benedikt and Lewkowitsch, *Oils, Fats, and Waxes*, p. 318.

21.—TOCHER^a TEST FOR SESAME OIL.

Dissolve 1 gram pyrogallol in 15 cc of concentrated hydrochloric acid. Mix this solution with 15 cc of oil in a separatory funnel and allow to stand for a minute. Draw off the aqueous layer and boil for five minutes. In the presence of sesame oil it becomes colored red by transmitted light and blue by reflected light.

22.—MICROSCOPICAL EXAMINATION.^b

Dissolve in a test tube from 2 to 5 grams of oil or fat in about 10 cc of ether, plug the test tube lightly with cotton and allow to stand 15 or more hours in a moderately cool place.

The most characteristic crystals are obtained when the crystallization proceeds slowly and at temperature of from 22° to 24° C. The first crop of crystals may be examined and the mother liquor separated and set aside for further crystallization.

In order to get rid of the oleins, Gladding^c has suggested the following:

Dissolve in an Erlenmeyer flask 5 grams of melted fat in 10 cc of absolute alcohol and 5 cc of ether, stopper with cotton and place in ice water for about one-half hour, until the more crystallizable portions of the fat have separated. The crystalline part is separated by filtration through a filter paper moistened with alcohol, and washed with the alcohol-ether mixture. After drying in the air for some time the crystals are dissolved from the paper by means of ether and then treated in the same way as described in the first method. When the crystals are ready to examine a drop is removed with a pipette, placed on a slide, a drop of cotton oil or olive oil added, and a cover slip pressed gently down.

III.—DAIRY PRODUCTS.

By J. A. LeCLERC,

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(A) MILK AND CREAM.

1.—GENERAL DISCUSSION.

There are three kinds of adulteration generally practiced with milk. First, addition of water, which is the simplest and the most common practice. Second, removal of fat or the removal of fat and addition of water. This double adulteration is used in order not to disturb the specific gravity. Third, the addition of preservatives, most commonly formaldehyde, boric acid, or borax.

The determinations ordinarily made in the examination of milk and cream are specific gravity, fat, total solids, solids not fat, and the detection of preservatives and coloring matter. The specific gravity alone is of little value in judging the purity of milk, owing to the fact that the increase of specific gravity produced by the removal of cream may be reduced by the addition of water. One of the most important considerations is the relation of the solids not fat to the fat. In milk it has been found that this ratio does not vary widely from 9:4.

2.—DETERMINATION OF TOTAL SOLIDS.^d

Heat at 100° C. to constant weight about 3 grams of milk in a tared platinum, aluminum, or tin dish^e of 5 cm diameter, with or without the addition of 15 to 30 grams of sand. Cool and weigh.

^aPharm. Journ. and Trans., 1891, 639. Chem. Zeit., Rep., 1891, 5, 15-33. Benedikt and Lewkowitsch, Oils, Fats, and Waxes, p. 319. Winton, Conn. Expt. Sta. Rept., 1900, pt. 2, p. 153.

^bU. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 4, p. 449. Gladding, Jour. Am. Chem. Soc., 1896, 18, 189. Wiley Prin. & Prac. Agri. Anal., vol. 3, pp. 345, 346. Winton, Report Conn. Expt. Sta., 1900, pt. 2, p. 145.

^cJour. Am. Chem. Soc., 1896, 18, 189.

^dU. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 54.

^eSee Appendix, p. 151.

3.—DETERMINATION OF FAT.

(a) OFFICIAL METHOD.^a

Dry about 5 grams of the sample on ignited asbestos in a Hofmeister schälchen or in a perforated metal cylinder (described by Babcock) and extract with ether in a continuous extraction apparatus.

(b) BABCOCK'S METHOD.^b

This method is commonly used where a large number of samples is to be examined. Owing to the general use of this method and its wide publication it is not deemed advisable to introduce its description here.

(c) GERBER'S METHOD.

Where only occasional samples are to be examined Gerber's acid butyrometer is found to give results comparable with those of the Babcock apparatus and is much more convenient. Directions accompany the apparatus.

4.—DETERMINATION OF SOLIDS NOT FAT.

Deduct the percentage of fat from the percentage of total solids.

5.—DETECTION OF GELATIN.^c

"An acid solution of mercuric nitrate is prepared by dissolving mercury in twice its weight of nitric acid of 1.42 specific gravity, and diluting this solution to 25 times its bulk by the addition of water. Ten cubic centimeters of the milk or cream to be examined are mixed with an equal volume of the acid mercuric nitrate solution, the mixture is shaken, and then 20 cc of water are added. The liquid is again shaken, allowed to stand five minutes, and filtered. In the presence of much gelatin the filtrate will be opalescent and can not be obtained quite clear. To a portion of the filtrate contained in a test tube an equal volume of a saturated aqueous solution of picric acid is added. A yellow precipitate will be produced in presence of any considerable amount of gelatin, while smaller amounts will be indicated by the cloudiness produced by the picric acid solution. In the absence of gelatin the filtrate obtained will be perfectly clear, and will be unaffected by adding picric acid."

6.—DETECTION OF FORMALDEHYDE.^d

To the milk to be tested add strong commercial sulphuric acid without mixing, and at the junction of the two liquids a violet or blue color will appear if the milk contains one or more parts per 10,000 of formaldehyde. This color is supposed to be given only when there is a trace of ferric chlorid or other oxidizing agent present.

Other methods of detecting formaldehyde are described on pages 79 and 107.

7.—DETECTION OF BORAX AND BORIC ACID.

Use methods described on page 110.

8.—DETECTION OF FOREIGN COLORS.

(a) LEACH'S METHOD.^e

Warm about 150 cc of milk in a casserole over the flame and add about 5 cc of acetic acid, after which slowly continue the heating nearly to the boiling point while

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 54.

^b Wis. Exp. Sta. Bul. No. 24, and U. S. Dept. of Agr., Div. of Chem., Bul. No. 28, p. 34-42.

^c Allen, Com. Org. Anal., 2d ed., Vol. IV, pp. 181-182.

^d See Appendix, p. 151.

^e Jour. Am. Chem. Soc., 1900, 22, 207.

stirring. Gather the curd, when possible, into one mass by the stirring rod, and pour off the whey. If the curd breaks up into small flecks, separate from the whey by straining through a sieve or colander. Press the curd free from adhering liquid, transfer to a small flask, and macerate for several hours (preferably over night) in about 50 cc of ether, the flask being tightly corked and shaken at intervals.

(1) *Detection of annatto (in the ether extract).*

Decant the ether extract into an evaporating dish, place on the water bath, and evaporate off the ether. Make the fatty residue alkaline with sodium hydroxid, and pour upon a very small wet filter while still warm. After the solution has passed through, wash off the fat from the filter with a stream of water and dry the paper. If, after drying, the paper is colored orange, the presence of annatto is indicated, confirmed by applying a drop of stannous chlorid solution, which, in presence of annatto, produces a characteristic pink on the orange-colored paper.

(2) *Detection of aniline orange (in the curd).*

The curd of an uncolored milk should be perfectly white after complete extraction with ether, as would also that of a milk colored with annatto.

If the extracted fat-free curd is distinctly dyed an orange or yellowish color, aniline orange is indicated. To confirm the presence of this color, treat a lump of the fat-free curd in a test tube with a little strong hydrochloric acid. If the curd immediately turns pink, the presence of aniline orange is assured.

(3) *Detection of caramel (in the curd).*

If the fat-free curd is colored a dull brown, caramel is to be suspected. Shake a lump of the curd, as in (b), with strong hydrochloric acid in a test tube and heat gently. The acid solution of the caramel-colored curd will gradually turn a deep blue, as would also the white, fat-free curd of an uncolored milk, while the curd itself does not change color. ^a

(b) LYTHGOE'S TEST FOR ANILINE ORANGE. ^b

Treat about 10 cc of the milk with an equal volume of hydrochloric acid (sp. gr. 1.20) in a porcelain casserole, and give the dish a slight rotary motion. If aniline orange is present in appreciable amount a pink color will at once be imparted to the curd particles as they separate out.

(B) BUTTER.

1.—GENERAL DISCUSSION.

The most common adulteration of butter is the substitution of fat other than butter fat. This is effected either by oleomargarine or by mixtures of oleomargarine and butter. Within the last few years a new product, called process or renovated butter, has been sold extensively as butter. The process of its manufacture is, briefly, as follows:

Poor and rancid butter is melted, the curd and brine are allowed to settle, the froth and scum skimmed off, after which the clear fat is drawn off and completely aerated so as to remove any unpleasant odors. Next, pure skimmed or whole milk is added and the mixture is thoroughly stirred so as to form a complete emulsion, and is

^a It should be noted that it is only when this blue coloration of the acid occurs in connection with a *brown-colored* curd, which itself does not change color, that caramel is to be suspected, as distinguished from the pink coloration produced at once under similar conditions by aniline orange. It is to be regretted that there are no such definite confirmatory tests for caramel as there are for annatto and aniline orange. See 19th An. Rep. Mass. State Board of Health (1887), p. 183.

^b Jour. Am. Chem. Soc., 1900, 22, 813.

finally sprayed into ice-cold water so as to give the product that granular appearance usually found in butter just from the churn. It is then treated and sold as butter. When this product, or its mixture with butter, is sold as butter it should be considered an adulteration. In addition to the special methods given below for the detection of process butter, the general methods described under Edible Fats and Oils must often be employed. Excessive amounts of water or of casein should be regarded as adulterations. Occasionally preservatives other than salt are added to butter, but they are generally the same as those found in milk.

2.—DETERMINATION OF WATER.^a

Place from 1.5 to 2.5 grams of the sample in a flat-bottomed dish having a surface of at least 20 square centimeters, and dry to constant weight at the temperature of boiling water.

The use of clean, dry sand or asbestos with the butter is admissible, and is necessary if a dish with round bottom be employed.

3.—DETERMINATION OF FAT.^a

(a) DIRECT METHOD.

Dry the butter on asbestos or sand to determine the water, and extract the fat by anhydrous alcohol-free ether. Evaporate the ether from the extract, heat to constant weight at the temperature of boiling water, and weigh.

(b) INDIRECT METHOD.

Dissolve the dry butter from the water determination in the same dish with absolute ether or with 76° C. petroleum ether. Then transfer the contents of the dish to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and wash until free from fat. Heat the crucible and contents at the temperature of boiling water until the weight is constant, and calculate the weight of fat from the data obtained.

4.—DETERMINATION OF REICHERT-MEISSL NUMBER.

Employ the official method of the association.^b

5.—DETERMINATION OF SAPONIFICATION VALUE.

Proceed as directed on page 26.

6.—THE WATERHOUSE TEST FOR OLEOMARGARINE.^c

Half fill a 100-cc beaker with sweet milk, heat nearly to boiling, and add 5 to 10 grams of butter or oleomargarine. Stir with a small rod, preferably of wood and about the size of a match, until the fat is melted. Then place the beaker in cold water and stir the milk until the temperature falls sufficiently for the fat to congeal. At this point the fat, if oleo, can easily be collected into one lump by means of the rod, while if butter it will granulate and can not be so collected. The distinction is very marked.

7.—SPECIAL TESTS FOR PROCESS BUTTER.^d

(a) FOAM TEST.^e

Heat 2 or 3 grams of the sample, either in a spoon or dish, over a free flame. True butter will foam abundantly, whereas process butter will bump and sputter,

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 43.

^b U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, pp. 44-46.

^c Jour. Am. Chem. Soc., 1901, 23, 200; U. S. Dept. Agr., Farmers' Bul. 131, p. 7.

^d See Appendix, p. 152.

^e Jour. Amer. Chem. Soc., 1900, 22, 150; U. S. Dept. of Agr., Farmers' Bul. No. 131.

like hot grease, without foaming. Oleo behaves like process butter, but chemical tests and the Waterhouse test described above will indicate whether the sample is oleo or butter, either genuine or process.

(b) APPEARANCE OF MELTED BUTTER.^a

Melt from 50 to 100 grams of butter or process butter at 50° C. The curd from the butter will settle, leaving above it a clear, supernatant fat. On the other hand the supernatant fat in the case of process butter does not assume that clear appearance, but remains more or less turbid.

(c) MICROSCOPIC EXAMINATION.^b

Place a bit of the butter or process butter on a glass slide, cover it and press into a thin film with cover glass. Examine immediately with a polarizing microscope magnifying from 100 to 140 diameters. When a selenite plate is placed between the slide and the lower nicol a normal butter will give a uniformly blue colored field, showing the absence of fat crystals. On the other hand, process butter gives a blue field, mottled with yellow.

8.—DETECTION OF ANNATO AND SAFFRON IN BUTTER—CORNWALL'S METHOD.^c

Five grams fat are dissolved in 50 cc of ether in a wide tube and the solution is vigorously shaken with 12 to 15 cc of a very dilute solution of potassium hydroxid, which must still be alkaline after it separates from the ether solution. It is allowed to stand a few hours, when the aqueous layer is drawn off, evaporated to dryness, and tested with sulphuric acid, which in the presence of annato gives first a blue or violet blue, changing quickly to green, and finally to brown.

Saffron which would be extracted at the same time acts differently when treated with sulphuric acid, not giving the green coloration.

The aqueous solutions, if not clear enough to use, must not be filtered, as the filter paper will take up large amounts of the color, but can be shaken up again with fresh portions of ether.

Martin^d uses carbon disulphid as a solvent instead of ether, which is just as satisfactory.

Genuine butters treated in this way give only a very slight trace of coloring matter.

9.—DETECTION OF ANILINE COLORS.^e

Follow methods described under Coloring Matter (p. 111 and following).

(C) CHEESE.

1.—GENERAL DISCUSSION.

There are two kinds of adulterations practiced with cheese. First, the use of fat other than milk fat, producing a product called filled cheese. Second, the removal of varying amounts of fat, producing skim cheese. The liquefied fats of swine or cattle intimately mixed with skim milk produce filled cheese; therefore the chemical methods for the examination of the fat of filled cheese are the same as are used for the detection of adulterated butter. Skim cheese is made from milk from which part or the whole of the fat has been removed; therefore the determination of fat and nitrogenous compounds will give a good indication as to whether the sample under examination is skim cheese.

^a Jour. Amer. Chem. Soc., 1900, 22, 327.

^b Jour. Amer. Chem. Soc., 1900, 22, 327.

^c Chem. News, vol. 1887, 55, 49; U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 1, p. 28.

^d Analyst, 1885, 10, 163.

^e See Appendix, p. 152.

2.—SELECTION OF SAMPLE.^a

When the cheese can be cut take a narrow wedge-shaped segment reaching from the outer edge to the center of the cheese. Cut this into strips and pass through a sausage-grinding machine three times. When the cheese can not be cut take the sample with a cheese trier. If only one plug can be obtained take it perpendicular to the surface of the cheese at a point one-third of the distance from the edge to the center and extending either entirely or only half way through it. When possible draw three plugs—one from the center, one from a point near the outer edge, and one from a point halfway between the other two. For inspection purposes reject the rind; but for investigations requiring the absolute amount of fat in the cheese include the rind in the sample. It is preferable to grind the plugs in a sausage machine, but when this is not done they are cut very fine and carefully mixed.

3.—SEPARATION OF FAT FOR EXAMINATION.^b(a) FIRST METHOD.^c

Cut about 300 grams of cheese into fragments the size of a pea. Treat with 700 cc of potassium hydroxid (50 grams per liter) at 20° C. in a large wide-necked flask, and promote the solution of casein by vigorous shaking. In from 5 to 10 minutes the casein will be dissolved and the fat will come to the surface in lumps. Collect the lumps of fat into as large a mass as possible by a gentle shaking to and fro. Pour cold water into the flask until the fat is driven up into the neck and remove it by means of a spoon. Wash the fat thus obtained with as little water as will remove the residue of the lye which it may contain. Experience has shown that the fat is not perceptibly attacked by the lye in this treatment. By this method the fat is practically all separated in a short time and is then easily prepared for chemical analysis by filtering and drying as directed in the official method.^d

(b) SECOND METHOD.

Grind the cheese by passing it through a meat-cutting machine. Transfer it to a large flask and pour warm water upon it, using 1 cc for every gram of cheese. Shake thoroughly and add sulphuric acid (sp. gr. 1.82 to 1.825) slowly and in small quantities, shaking after each addition of acid. The total amount of acid used should be the same as the amount of water used. Remove the fat, which separates after standing a few minutes, by means of a separatory funnel, wash it free from acid, filter, and dry.

4.—DETERMINATION OF WATER.^e

Place from 2 to 5 grams of cheese in a weighed platinum dish containing a small quantity of porous material such as ignited asbestos or sand, to absorb the fat which may run out of the cheese. Heat in a water-jacketed bath for ten hours and weigh; the loss in weight is considered as water. Or, if preferred, place the dish in a desiccator over concentrated sulphuric acid and dry to constant weight. Renew the acid when the cheese has become nearly dry.

5.—DETERMINATION OF FAT.^e

Cover the perforations in the bottom of the extraction tube with dry asbestos, and on this place a mixture containing equal parts of anhydrous copper sulphate and

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 55.

^b See also Appendix, p. 152.

^c U. S. Dept. of Agr., Div. of Chem., Bul. 51.

^d U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 44, 3 (a).

^e U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 56.

pure dry sand to the depth of about 5 cm. packing loosely. Cover the upper surface of this material with a film of asbestos and place on it from 2 to 5 grams of the sample of cheese. Place the tube in a continuous extraction apparatus and treat for five hours with anhydrous ether. Remove the cheese and grind to a fine powder with pure sand in a mortar. Replace the mixed cheese and sand in the extraction tube, wash the mortar free of all matters with ether, add the washings to the tube, and continue the extraction for ten hours.

6.—DETERMINATION OF NITROGENOUS COMPOUNDS.

Make a determination of nitrogen by the Kjeldahl or the Gunning method, using about 2 grams of cheese, and multiply the percentage of nitrogen found by 6.25.

IV.—CEREAL PRODUCTS.

By A. MCGILL,

Chemist of Inland Revenue Laboratory, Ottawa, Canada.

It has been found impossible to prepare the report on this subject this year. The heading has been inserted here to preserve its proper order.

V.—INFANT AND INVALID FOODS.

By H. W. WILEY,

Chief of Bureau of Chemistry, United States Department of Agriculture.

1.—GENERAL DISCUSSION.

Under this head are included all prepared foods of every description, which are intended especially for the use of infants and invalids.

It is evident that foods for infants should be as nearly as possible similar in character to the natural food, viz, healthy human milk. All modified milk, of the cow and other animals, intended for infants, would be included in this class. If these milks be evaporated, they would differ from the original sample only in the loss of water, provided the evaporation be carried on in vacuum at low temperature. Many of the foods advertised for the use of infants, however, contain starch and other matters not usually found in healthy human milk. The number of foods advertised for the use of infants is legion. Many of them are "predigested," that is, they have the proteid matter reduced to the form of more or less soluble protein and the starchy matters converted more or less completely into soluble carbohydrates.

Under the head of infant and invalid foods should also be considered the various products in which nitrogenous bodies are the most important ingredients.

Infant foods may be divided into two classes: First, milk of cows and other animals modified to resemble more or less completely healthy human milk; and, second, the foods in which carbohydrates are the predominating element. In this connection it may be stated that preparations of this nature are ordinarily used with milk, and may be considered in a sense as substitutes for milk sugar.

The modification of milk consists as a rule in diminishing the proportion of the protein matter or casein and increasing that of milk sugar and fat. By chemical and other treatment, the ordinary average milk of the cow may thus be modified so as to resemble more or less completely the average healthy human milk. It is planned to examine a large number of infant and invalid foods during the coming year, and for that reason the writer had intended to defer his report until the completion of the work mentioned. So many inquiries have recently been received regarding this subject, however, that it was thought best to prepare for this bulletin a brief summary of methods to be employed with references to a previous publication of the writer's describing them.

2.—METHODS OF DETERMINATION.

Determinations in this case should consist of:

(a) WATER.

The water can be determined in the usual manner by evaporating a small quantity of the material in a flat dish, so that the film of solid matter may not be too thick. This is preferably done in vacuo or in an atmosphere of inert gas. See Principles and Practice of Agricultural Analysis, vol. 3, pages 13 and following; also Department of Agriculture, Division of Chemistry, Bulletin No. 46 revised, pages 27 and 43.

(b) ASH.

Burn the dried sample at a low red heat, preferably in a muffle. See Principles and Practice of Agricultural Analysis, vol. 3, pages 36 and following; also Bulletin 46 revised, page 23.

(c) FAT.

Determine by one of the methods given in Principles and Practice of Agricultural Analysis, vol. 3, pages 480 and following, and Bulletin 46 revised, page 54.

(d) SUGARS.

The lactose may be determined both by reduction of copper salts and by optical processes. See Principles and Practice of Agricultural Analysis, vol. 3, pages 275 and following; and Bulletin 46 revised, pages 40 and following.

(e) ADDED SUCROSE.

Use method of Bigelow and McElroy, Principles and Practice of Agricultural Analysis, vol. 3, page 296; or that of Stokes and Bodmer, Analyst, 1885, 10, 62.

(f) PROTEIN.

Use methods described in Principles and Practice of Agricultural Analysis, vol. 3, pages 504 and following, for total protein and separation of protein matters; also Bulletin 46 revised, pages 54 and 55.

3.—CONDENSED MILK.

Mix the entire contents of the can, transfer 250 grams to a liter flask, dissolve in water and make the solution up to the mark. The solution should then be treated for various constituents as under dairy products on aliquot parts of the contents of the flask.

4.—CARBOHYDRATE FOODS.

Another class of foods for infants and invalids, as intimated above, is chiefly composed of carbohydrate bodies. These foods should be examined microscopically to determine, if possible, the origin and character of the starch. The water and ash should be determined by the usual methods. The quantity of starch unchanged should be determined. See Principles and Practice of Agricultural Analysis, vol. 3, pages 201 and following; and Bulletin 46 revised, page 25.

(a) DEXTRIN.

This can be determined in the solution of the bodies after the fermentation of other sugars. Dextrin can then be determined by its opticity or by precipitation with alcohol. See Principles and Practice of Agricultural Analysis, vol. 3, pages 287 and following.

(b) DEXTROSE.

Determine dextrose by the methods given in Principles and Practice of Agricultural Analysis, vol. 3, pages 287 and following.

(c) INVERT SUGAR.

See Principles and Practice of Agricultural Analysis, vol. 3, pages 161, 162, 257 and following.

(d) MALTOSE.

For separation from dextrin and dextrose, see Principles and Practice of Agricultural Analysis, vol. 3, pages 287 and following; for estimation, see pages 165 and following.

The important point to be determined in these foods is the extent to which the so-called predigestion has been carried, and this is done by ascertaining the condition of the carbohydrate and proteid bodies. Attention should also be given to the nature of any ferments which have been employed in effecting the predigestion, or acids, if such have been used. Further, these foods should be examined for preservatives, which are sometimes added when the samples are in a liquid state, or are perishable in character.

VI.—SACCHARINE PRODUCTS.

By ALBERT E. LEACH,

Analyst of State Board of Health, Boston, Mass.

1.—GENERAL DISCUSSION.

This class of food products, from their nature and composition, is so closely allied to sugar and starch (methods for which have been already so fully studied by this association) that little remains of a purely distinctive character in their examination. Being, furthermore, composed almost exclusively of carbohydrates, it is doubtful if, as a rule, much attention need be given to the determination of nitrogen or fatty constituents, which occur in very minute quantities only, and chiefly in chocolate and flavoring material, or in the eggs and butter that enter incidentally into the manufacture of confectionery.

2.—PREPARATION OF THE SAMPLE.

(a) MOLASSES AND SIRUP.

Insure a homogenous mixture by stirring with a rod till any crystallized sugar is evenly distributed throughout the mass.

(b) HONEY.

Treat the strained honey as in the case of molasses, 2 (a).

In the case of comb honey, cut across the top of the comb, if sealed, and separate completely from the comb by straining through a 40-mesh sieve.

If the honey has become wholly or in part solidified by crystallization, use a gentle heat on a closed water bath to restore it to fluid form.

(c) CONFECTIONERY.

(1) *Products of practically uniform composition throughout.*

(a) *Lozenges and other pulverizable products.*—Grind in a mortar or mill to a fine powder.

(b) *Semiplastic, sirupy, or pasty products.*—Weigh 50 grams of the sample into a 250-cc graduated flask, mix thoroughly or dissolve, if soluble, in water and fill to the mark. Be sure that the solution is uniform, or, if insoluble material is present, that it is evenly mixed by shaking before taking aliquot parts for the various determinations.

(2) *Confectionery in layers or sections of different composition.*

When it is desired to examine the different portions separately they should be separated mechanically with a knife when possible, and treated as directed under (1).

(3) *Sugar-coated fruit, nuts, etc.*

In case of a saccharine coating enclosing fruit, nuts, or any less readily soluble material, dissolve or wash off the exterior coating in water, which may, if desired, be evaporated to dryness for weighing, and proceed as in (1).

(4) *Brandy drops and similar preparations.*

In case of a hard exterior coating enclosing a sirup or fluid which it is desired to examine, puncture the outer coating with a knife and pour out the fluid, using a sufficient number of the "drops" to yield enough fluid for examination (see page 49, sec. 14).

(5) *Candied or sugared fruits.*

Proceed as directed under Preserves and Canned Fruits, page 75.

3.—DETERMINATION OF TOTAL SOLIDS.

(a) *MOLASSES, SIRUPS, AND HONEY.*

(1) *By direct determination.*

Weigh 20 grams into a 100-cc graduated flask, dissolve in water and make up to the mark. Insure a uniform solution by shaking. Measure 10 cc of this solution into a tared platinum dish containing about 5 grams of freshly ignited, finely divided asbestos fiber, and dry to constant weight at 70° in vacuo or in a McGill oven (see footnote on page 76).

(2) *By calculation from specific gravity.^a*

Weigh 25 grams of the sample into a 100-cc graduated flask, dissolve in water and make up to the mark. Determine the specific gravity of the diluted solution by means of a pycnometer or Westphal balance. Ascertain from Table VI the percentage by weight of solids corresponding to the specific gravity of the diluted solution and calculate the total solids in the original sample by the formula: Solids in original sample = 4DS, D being the specific gravity of the diluted solution and S the per cent of solids in the diluted solution.

(b) *CONFECTIONERY.*

(1) *Lozenges and other pulverizable products.*

Weigh from 2 to 5 grams of the powdered sample in a tared platinum dish and dry to constant weight at 70° C. in vacuo or in the McGill oven.

(2) *Semiplastic, sirupy, or pasty products.*

Measure 25 cc of the 20 per cent solution, or mixture—2 (c) (1) (b)—into a tared platinum dish containing asbestos fiber and proceed as in 3 (a) (1).

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 28.

4.—DETERMINATION OF ASH.

Weigh out from 5 to 10 grams of molasses, honey, or sirup, or weigh 5 grams of the pulverizable confectionery, or measure 50 cc of the 20 per cent solution—2 (c) (1) (b)—into a tared platinum dish; evaporate to dryness on the water bath and burn slowly and cautiously over a low flame. After frothing has ceased, increase the flame and ignite to a white ash at a low, red heat.

In igniting saccharine products, frothing may be largely held in check by directing the flame at first down upon the mass from above instead of from under the dish, as ordinarily, until the material is well charred.

5.—EXAMINATION FOR MINERAL ADULTERANTS.

(a) REDUCING TO ASH.

Comparatively large quantities of saccharine products may be readily and quickly reduced to an ash for mineral examination without the troublesome frothing that ordinarily ensues in igniting at once with a free flame by proceeding as follows:^a

Mix 100 grams of molasses, sirup, or honey, or of the confectionery solution (2 (c) (1) (b)) evaporated to sirupy consistency, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating dish. An electric current is then passed through it while stirring, by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod, with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4.^b In from ten to fifteen minutes the mass is reduced to a fine, dry char, which may then be readily burnt to a white ash in the original dish over a free flame or in a muffle.

If an electric current is unavailable, treat in a large porcelain evaporating dish 100 grams of the saccharine solution to be ashed, which should be evaporated to sirupy consistency if not already such, with sufficient concentrated sulphuric acid to thoroughly carbonize the mass, after which ignite in the usual manner.

Among the suspected adulterants to be looked for in the ash are salts of tin, used in molasses to bleach or lighten the color, and mineral pigments such as chromate of lead in yellow confectionery, and oxide of iron, the latter being commonly used as an intensifier of or substitute for the natural color of chocolate.

(b) DETERMINATION OF TIN IN MOLASSES^c AND OTHER SACCHARINE PRODUCTS.^d

Fuse the ash from a weighed portion of the sample with sodium hydroxid in a silver crucible, dissolve in water and acidulate with hydrochloric acid;^e filter and precipitate the tin from this solution with hydrogen sulphid; wash the precipitate on a filter and dissolve it in an excess of ammonium sulphid. Filter this solution into a tared platinum dish and deposit the tin directly in the dish by electrolysis, using a current of 0.05 ampere. This current may be readily reduced from an ordinary 110-volt street circuit by means of a series of lamps, or a rheostat may readily be improvised for this purpose, consisting of a long, vertical glass tube, sealed at the bottom, containing a column of dilute acid through which the current passes, the

^a Leach. 32d An. Rept. Mass. State Board of Health. (1900.) p. 653. Reprint, p. 37.

This method is preferred to the ordinary method of heating with sulphuric acid, especially in case of molasses, because, if properly manipulated, it so quietly comes into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

^b Modified from method of Budde & Schou for determining nitrogen electrolytically. *Ztschr. anal. Chem.*, 1899, **38**, 345.

^c Leach. 31st An. Rept. Mass. State Board of Health, 1899, p. 625; Hilger & Laband, *Ztschr. für Untersuchung der Nahr.- u. Genuss.*, 1899, **2**, 795.

^d This method is applicable also to condensed milk, canned goods, etc.

^e See Methods for the examination of canned vegetables, p. 52.

resistance being changed by varying the length of the acid column contained between two electrodes immersed therein, one of which is movable.^a

6.—DETERMINATION OF ETHER EXTRACT IN CONFECTIONERY.

Measure 25 cc of the 20 per cent mixture or solution—2(c)(1)(b)—into a very thin, readily frangible glass evaporating shell (*Hoffmeister's Schälchen*), containing 5 to 7 grams of freshly ignited asbestos fiber; or, if impossible to thus obtain a uniform sample, weigh out 5 grams of the mixed, finely divided sample into a dish, and wash with water into the asbestos in the evaporating shell, using, if necessary, a small portion of the asbestos fiber on a stirring rod to transfer the last traces of the sample from dish to shell. Dry to constant weight at 100°, after which cool, wrap loosely in smooth paper, and crush into rather small fragments between the fingers, carefully transferring the pieces with the aid of a camel's hair brush to an extraction tube or a Schleicher and Schull cartridge for fat extraction. Extract with anhydrous ether or with petroleum ether in a continuous extraction apparatus for at least 25 hours. Transfer the solution to a tared flask, evaporate off the ether, dry in an oven at 100° C. to constant weight, and weigh.

Unless the ether is absolutely anhydrous, sugar will be dissolved. Ether which gives off hydrogen when treated with metallic sodium is unfit to use without purification. To purify it, let it stand for some time with calcium chlorid in the container, then pour off and distill over metallic sodium.

If petroleum ether is employed, it should be purified by fractional distillation so that it boils between 45° and 60° C. and leaves absolutely no residue.

7.—DETERMINATION OF PARAFFIN IN CONFECTIONERY.

Add to the ether extract in the flask as above obtained, 10 cc of 95 per cent alcohol and 2 cc of 1:1 sodium hydroxid solution, connect the flask with a reflux condenser, and heat for an hour on the water bath or until saponification is complete. Remove the condenser, and allow the flask to remain on the bath till the alcohol is evaporated off and a dry residue is left. Treat the residue with about 40 cc of water and heat on the bath, with frequent shaking, till everything soluble is in solution. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would come down with the paraffin, but the amount would be so insignificant that except in the most exacting work it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting point and specific gravity, and by subjecting it to examination in the butyro-refractometer. The melting point of paraffin is about 54.5° C.; its specific gravity at 15.5° is from 0.868 to 0.915, and on the refractometer (Zeiss's scale) the reading at 65° C. is from 11 to 14.5.

8.—DETERMINATION OF NITROGEN.

Use 5 grams of the sample for this determination, and follow the details of the regular Gunning method.^b

9.—DETERMINATION OF STARCH IN CONFECTIONERY.^c

Measure gradually 25 cc of the 20 per cent solution or uniform mixture (2 (c) (1) (b)) into a hardened filter or Gooch crucible, or transfer by washing 5 grams of the finely powdered substance to the filter or Gooch, and allow the residue on the filter to become air-dried. Extract with 5 successive portions of 10 cc of ether, then wash

^a Wiley, Principles of Agricultural Analyses, vol. 3, p. 152.

^b U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 16.

^c U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 25.

with 150 cc of 10 per cent alcohol, and finally with 20 cc of strong alcohol. Transfer the residue to a large flask and boil gently for 4 hours with 200 cc of water and 20 cc hydrochloric acid (specific gravity 1.125), the flask being provided with a reflux condenser. Cool, neutralize with sodium hydroxid, add 5 cc of alumina cream, and make up the volume to 250 cc with water. Filter, and determine the dextrose in an aliquot part of the filtrate by Allihn's method, as directed in 13, page 49. The weight of the dextrose multiplied by 0.9 gives the weight of the starch.

10.—POLARIZATION.

(a) MOLASSES.

Dissolve the normal weight of the sample (26.048 grams for the Schmidt and Haensch polariscope) in water in a 100-cc graduated flask, add an excess of lead subacetate solution,^a and fill to the mark; shake to insure uniform solution, filter and polarize in a 100-mm tube, multiplying the reading by 2 for the direct polarization.

To 50 cc of the filtrate add 5 cc of concentrated hydrochloric acid. Heat slowly to 68° and cool. Polarize in the same tube at the same temperature as before, add 10 per cent to the reading, and multiply by 2 for the invert polarization.

The short tube (100-mm) is preferred for polarizing molasses not only on account of the more or less deep color of the clarified solution, but also because a molasses sample containing considerable commercial glucose would not read within the scale limits if the 200-mm tube were employed.

It sometimes happens, especially with molasses containing much glucose sirup, that it is impossible to obtain a clear filtrate after clarification with lead subacetate, or that the filtrate, at first clear, clouds up too quickly to admit of a satisfactory reading. In such cases weigh out a fresh portion of the sample, dilute, and add first the lead subacetate solution and then enough sodium sulphate or common salt to precipitate the excess of lead. Afterwards fill to the mark and proceed in the regular manner.

For medium or light-colored grades of molasses which yield but a small precipitate with lead subacetate, the above method of simple polarization both direct and invert gives results sufficiently accurate for ordinary work. For dark-colored or "black strap" molasses, or wherever extreme accuracy is required, employ the double-dilution method—10 (c).

(b) HONEY, MAPLE SIRUP, AND WATER-SOLUBLE CONFECTIONERY.

Follow directions given under "molasses," 10 (a), except that alumina cream^b is employed in excess as a clarifier instead of subacetate of lead.

(c) CONFECTIONERY CONTAINING STARCH OR INSOLUBLE MATTER.

Employ the double-dilution method,^c thus making due allowance for the volume of the precipitate. Take half the normal weight of the sample and make up the solution to 100 cc, using the appropriate clarifier (subacetate of lead for dark-colored confectionery or molasses, and alumina cream for light-colored confectionery and honey). Take the normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each in the usual manner and obtain the invert readings of the two.

The true direct polarization of the sample is the product of the two direct readings divided by their difference. The true invert polarization is the product of the two invert readings divided by their difference.

^a See footnote, page 84.

^b Prepare by dividing a cold saturated aqueous solution of alum into two unequal portions, to the larger of which add a slight excess of ammonium hydroxid. Then add by degrees the remaining portion to a faint acid reaction. U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 39.

^c Wiley & Ewell, Analyst, 1896, 21, 184.

11.—DETERMINATION OF CANE SUGAR.

Use Clerget's formula:

$$S = \frac{(a-b) 100}{144 - \frac{t}{2}}$$

where S =per cent of cane sugar, a =direct polarization, b =invert polarization, t =temperature.

12.—DETERMINATION OF COMMERCIAL GLUCOSE IN MOLASSES, SIRUPS, AND HONEY.^a

As to preliminary indications of the presence of commercial glucose in these products, a sample of molasses of light color whose normal weight made up to 100 cc and polarized in a 200-mm tube shows a reading much in excess of 60° on the cane-sugar scale is almost sure to contain commercial glucose, while a dark-colored sample of molasses should, if pure, polarize considerably below 50° C. A sample of maple sirup which polarizes much in excess of 65° C on the cane-sugar scale is to be suspected of containing commercial glucose, while a sample of honey that polarizes to the right of the zero point is apt to be adulterated either with cane sugar or commercial glucose or both. If any of these products show an invert reading much to the right of the zero point, commercial glucose is almost sure to be present.

It is manifestly impossible to determine with absolute accuracy the amount of commercial glucose present by reason of the varying amount of dextrine, maltose, and dextrose present in the adulterant. It is possible, however, in molasses and maple sirup, wherein the amount of invert sugar is so small as not to appreciably affect the result, to estimate approximately the amount of commercial glucose by the following formula:

$$G = \frac{(a-S) 100}{175}$$

where G =per cent of commercial glucose, a =direct polarization, S =per cent of cane sugar.

In honey, which is composed largely of invert sugar, much closer results are attained by first inverting the sample and obtaining the polariscopic reading at 87° in a tube surrounded by hot water. This reading divided by 175 gives the approximate percentage of commercial glucose in the sample.

A large number of samples of commercial glucose have been procured by the department of food and drug inspection of the Massachusetts Board of Health directly from various manufacturers of compound or adulterated honey, molasses, and sirup, to ascertain the grade used by them for this purpose. As a result of this investigation, it has been found that the grade best adapted by its consistency for admixture with these products, and, indeed, the grade largely, if not universally, used for this purpose, has a density of about 42° Beaumé and polarizes on the cane-sugar scale at or about 175° (26.048 grams made up to 100 cc and polarized in a 200-mm tube with the Schmidt & Haensch instrument). From repeated experiments made in the writer's laboratory on mixtures containing known proportions of commercial glucose, 175 has been adopted as the most satisfactory factor and has been found to give a very close approximation.^b

^a Leach, 32d An. Rept. Mass. State Board of Health, 1900, p. 658. Reprint, p. 42.

^b Among the samples of commercial glucose examined were several obtained from manufacturers of compound jellies and jams, and the examination of these would seem to show that the grade used mostly for this purpose polarizes at or about 150° C. If this is verified, 150 instead of 175 should be used in the above formula when applied to jellies and jams. The effect of high temperatures employed in the preparation of this class of goods should not be lost sight of, a factor that does not enter in to disturb the application of the method to adulterated molasses, sirups, and honeys which are mixed in the cold.

For chewing gum a grade of commercial glucose is used polarizing at about 185°.

For confectionery, as might be expected, from the wide variation in the character and consistency of candies, there is little uniformity in the grade of commercial glucose employed, so that it is not possible as in the case of molasses and honey to calculate the amount present. Nor is it so essential, in view of the fact that commercial glucose is rarely regarded as an adulterant of confectionery.

13.—DETERMINATION OF REDUCING SUGARS (ESTIMATED AS DEXTROSE).

Treat 5 grams of molasses, sirup, or honey, or 25 cc of the 20 per cent solution or mixture (2 (c) (1) (b)) or 5 grams of the powdered confectionery, with water in a 100-cc graduated flask, using 2 to 5 cc, of lead subacetate solution in the case of molasses or sirup, and 5 cc of alumina cream in the case of honey or confectionery. Make up to 100 cc, filter, take an aliquot part of the filtrate (25 to 50 cc), and make this up to 100 cc, the amount taken being such that when diluted the solution will contain not more than one per cent of dextrose. If lead subacetate has been used to clarify, add to the aliquot part taken, and before dilution, enough sodium sulphate to precipitate the excess of lead, then filter, and make up to the 100-cc mark.

Add 30 cc of Fehling's copper solution^a to 30 cc of Fehling's alkaline tartarate solution^b in a 250-cc Erlenmeyer flask.^c Add 25 cc of the sugar solution (which must not contain more than 1 per cent of reducing sugar) with a burette, heat to boiling and boil exactly 2 minutes. Separate the precipitate as quickly as possible by filtering, with the aid of vacuum, through a layer of asbestos about 1 cm thick in a Gooch crucible (which with the asbestos has previously been ignited, cooled, and weighed), washing the cuprous oxide precipitate with boiling distilled water till the wash water ceases to be alkaline.

To prepare the asbestos, first boil it with nitric acid (sp. gr. 1.05 to 1.10), washing out the acid with hot water, then boil with a 25 per cent solution of sodium hydroxid and finally wash out the alkali with hot water. Keep the asbestos in water in a wide-mouthed flask or bottle, and transfer it to the Gooch by shaking it up in the water and pouring it quickly into the crucible while under suction.

Dry the Gooch with its contents in the oven, and finally heat it at dull redness for fifteen minutes. Transfer to the desiccator, cool, and weigh quickly as cupric oxid. A platinum Gooch may safely be used. If a porcelain Gooch is employed, extra precautions are necessary in heating to avoid cracking. With porcelain use a muffle.

Or, wash with alcohol and ether, dry for 20 minutes at 100° C. and weigh as cuprous oxid. In either case ascertain the weight of reducing sugar, in terms of dextrose, from Table VIII.

Or, the copper may be determined from the cuprous oxid in accordance with the official methods.^d

14.—DETERMINATION OF ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").^e

Open each drop by cutting off a section with a sharp knife and collect in a beaker the sirup of from 15 to 25 of the drops, which will usually yield from 30 to 50 grams of sirup. Strain the sirup into a tared beaker through a perforated porcelain filter

^a 34.639 grams $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, dissolved in water and diluted to 500 cc.

^b 173 grams of Rochelle salts and 125 grams of potassium hydroxid dissolved in water and diluted to 500 cc.

^c Defren, Jour. Am. Chem. Soc., 1896, 18, 749.

^d U. S. Dept. of Agr., Div. of Chem., Bul. 46, p. 37.

^e Thirty-second An. Rep. Mass. Board of Health, 1900, p. 757. Reprint, p. 41.

plate in a funnel to separate from particles of the inclosing shell, and ascertain the weight of the sirup. Dilute with half its volume of water and determine alcohol as directed on page 82.

15.—DETECTION OF COLORING MATTER.

Proceed as directed under Coloring matters (p. 111 and following).

VII. CANNED VEGETABLES.

By L. S. MUNSON,

Bureau of Chemistry, U. S. Department of Agriculture.

1.—GENERAL DISCUSSION.

In the investigation of canned vegetables, the proximate analysis is, as a rule, of little value in determining quality; much more depends upon the size, age, and fresh and healthy condition of the vegetables at the time of canning, and the treatment during and subsequent to the processes of canning. Hence, methods for the proximate analysis have been given minor consideration to means of determining the quality of the various classes of vegetables and the detection of different forms of adulterants. Much still remains to be done with this class of food, as the time at the disposal of the writer was not sufficient to investigate thoroughly the various problems that presented themselves.

2.—MACROSCOPIC EXAMINATION.

A careful macroscopic examination is often of material value in detecting inferior quality with certain classes of vegetables. Upon opening a can, carefully note the appearance of the contents as to quality, color, and size. Any undue corrosion, or blackening of the walls of the can, should also be observed. With mushrooms and capers, no further examination is necessary, as a rule, except the detection of sulphites in the former.

The most common form of mushrooms found upon the market is *Agaricus campestris*, although different varieties of *Boletus* are occasionally found. The latter are particularly susceptible to attack by larvæ and, except in a fresh state, are seldom free from them. These larvæ may readily be seen with the naked eye, or by use of a small hand lens. Many of the mushrooms on the market are of inferior quality, and consist largely of old and broken fragments of tops and stems; occasionally diseased fungi are to be found in the inferior grades. Owing to the nature of this vegetable, only the fresh, healthy specimens should be passed as edible.

Capers are the flower buds of *Cappares spinosa* and, so far as known, are but little liable to adulteration. Owing to their green color, it is always advisable to make a qualitative test for copper.

Olives are to be judged entirely by general appearance and by taste. Gherkins and mixed pickles, while not strictly under this class of foods, are considered here along with olives for the sake of completeness; these also are to be judged largely macroscopically and by taste. The use of copper with this class is of frequent occurrence to produce the bright green color; with mixed pickles, where mustard is used, turmeric is frequently added as a coloring agent. It is also advisable to test for aniline dyes where turmeric is not detected.

3.—PREPARATION OF THE SAMPLE.^a

Weigh the full can; open, pour off the liquid portion, and reweigh the can; then empty out the solid contents of the can and weigh again. From these weights estimate the percentage of liquid and solid contents. By this means, any undue propor-

^a U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 8, p. 1027.

tion of liquor, owing to excess of water added, will be detected. Then thoroughly grind the entire contents of the can, either in a mortar or by means of a food chopper; mix thoroughly and preserve in a glass-stoppered bottle for analysis. Unless the analysis is to be completed within a reasonably short time it is best to dry the entire sample after the determination of moisture is made. After thorough drying, the material is allowed to stand exposed to the air for several hours, or until it has become air dry. A second moisture determination is necessary with this procedure.

4.—PROXIMATE ANALYSIS.

For methods of proximate analysis, see Bulletin 13, part 8, U. S. Department of Agriculture, Division of Chemistry, page 1028.

5.—DETECTION OF SACCHARIN.

Saccharin is quite extensively used in canned sweet corn as a sweetening agent. For its detection add from 25 to 40 cc of water to about 20 grams of the sample; macerate and strain through muslin; acidify with 2 cc of sulphuric acid (1 to 3) and extract with ether. Separate the ether layer, allow the ether to evaporate spontaneously, and take up the residue with water. If saccharin be present its presence will be indicated by the sweet taste imparted to the water. To confirm this test add from 1 to 2 grams of sodium hydroxid, and place the dish in an oil bath. Maintain the temperature of the oil at 250° C. for twenty minutes, when the saccharin will be converted into salicylic acid. After cooling and acidifying with sulphuric acid extract in the usual way and test for salicylic acid. This test, of course, presupposes the absence of salicylic acid in the original sample. If salicylic acid is present in the original sample it must be removed before making the test for saccharin.

6.—DETERMINATION OF SULPHITES.

Sulphites are largely used with certain classes of vegetables, notably corn and asparagus, as a bleaching agent; they may also find use with this class of foods as a preservative.

(a) DISTILLATION METHOD.

For their determination place 50 grams of the material in a distilling flask, add about 5 cc of a saturated solution of glacial phosphoric acid, and proceed with the distillation and the titration of the sulphite as directed on p. 90. Where only a qualitative test is desired, take the first few cubic centimeters of the distillate, add a slight excess of iodine solution, boil to expel the excess of iodine, then acidify with hydrochloric acid and add barium chlorid solution. This test is very delicate and is easily applied.

(b) REDUCTION METHOD.^a

To about 25 grams of the sample placed in a 200-cc Erlenmeyer flask add some pure zinc and several cubic centimeters of hydrochloric acid. In the presence of sulphites, hydrogen sulphid will be generated and may be tested for with lead paper. Traces of metallic sulphids are occasionally present in vegetables, and by the above test will indicate sulphites. Hence positive results obtained by this method should be verified by the distillation method.

It is always advisable to make the quantitative determination of sulphites, owing to the danger that the test may be due to traces of sulphids. A trace is not to be considered sufficient either as a bleaching agent or as a preservative.

7.—DETECTION OF PRESERVATIVES.

See methods given under Preservatives (p. 107, and following).

^a Dept. of Agr., Div. of Chem., Bul. 13, pt. 8, p. 1032.

8.—DETECTION OF COLORING MATTERS.

(a) IN TOMATOES^a AND CATSUPS.

Most of the coloring matter used in tomatoes is either of coal-tar origin or cochineal, and the general methods given under Coloring Matter (p. —) may be applied in these cases. Extract the color from the dried pulp with ordinary alcohol after acidifying with hydrochloric acid and filter. Eosin gives a characteristic fluorescent filtrate. Dilute the filtrate with water, extract with amyl alcohol and dye. Cochineal if present is in the form of a lake and will require strong hydrochloric acid to decompose it. After extraction with amyl alcohol it may be tested with uranium acetate (p. 120).

(b) IN PEAS, BEANS, GHERKINS, ETC.

Copper salts are most commonly employed in this class of goods, although it is said that zinc is occasionally used. For the qualitative detection, ash from 15 to 20 grams of the sample, either with or without previous treatment with concentrated sulphuric acid (see Heavy Metals below), transfer the ash to a beaker and treat with nitric acid; filter, make the filtrate alkaline with ammonia, and if a precipitate forms filter again. Copper will be indicated by the blue color of the filtrate. If further test is desired acidify with acetic acid, and add potassium ferro-cyanide. Red coloration or precipitate verifies the test.

(c) IN MIXED PICKLES, ETC.

Turmeric is frequently used and may be identified by the method given under Coloring Matter (p. 120).

9.—DETERMINATION OF TOTAL AND VOLATILE ACIDITY.

It is occasionally desirable to determine total acidity in tomatoes and catsups, and volatile acidity in the latter. For this purpose use methods described under Fermented and Distilled Liquors (p. 83). Express fixed acids as citric; one 1 cc of decinormal alkali equals .0070 gram of citric acid. Express volatile acids as acetic; 1 cc of decinormal alkali equals .0060 gram of acetic acid.

10.—DETERMINATION OF HEAVY METALS.

Owing to the almost universal presence of tin, the frequent occurrence of lead and zinc, and the extensive use of copper as a coloring agent in this class of food materials, the determination of heavy metals is of particular value. The method described by Allen^b and modified by Bigelow and Munson, has been used in the laboratory of the Bureau of Chemistry for the determination of heavy metals in canned meats, and may be applied as well to vegetables. Since the work on canned meats, however, the writer has worked out a method that for accuracy and ease of manipulation is preferred to the modified Allen's method.

(a) ALLEN'S METHOD, MODIFIED BY BIGELOW AND MUNSON.^c

Treat 100 grams of the moist material, or 25 grams of the dried material, with about 5 cc of concentrated sulphuric acid and 2 cc of nitric acid. After foaming has ceased add 3 grams of magnesium oxid and mix thoroughly. Then ignite over a Bunsen burner or, preferably, in a muffled furnace, until thoroughly charred. Grind in a mortar, and again ignite to complete combustion. The addition of a few drops of nitric acid may be necessary toward the end to complete the operation. Add

^a Girard and Dupré. *Analyses des matières alimentaires*, etc.

^b Allen's *Com. Organic Anal.* 3d ed. Vol. IV, p. 299.

^c *Jour. Amer. Chem. Soc. Proc.* 1900, 22, 32.

about 50 cc of hydrochloric acid (1:3) and heat to boiling or upon a steam bath for a half hour. Nearly neutralize the acid with sodium hydroxid dilute to 150 cc with water, precipitate with hydrogen sulphid, and filter, after heating for a few moments upon a steam bath to facilitate the separation of the precipitated sulphids. Dry the precipitate and insoluble ash residue, and then fuse in a porcelain crucible with a mixture consisting of one gram each of sodium carbonate, potassium carbonate, and sulphur. Dissolve the fused mass with hot water and filter. Sulphids of lead and copper remain upon the filter. Acidify the filtrate with acetic acid to precipitate the tin sulphid. Collect the tin sulphid upon a filter. Wash thoroughly, and then dissolve by the aid of heat in a concentrated solution of ferric chlorid. The reduced iron salt is then titrated with potassium dichromate.^a One cc of decinormal potassium dichromate equals 0.00295 grams of tin. The determination of the tin by igniting and weighing as stannic oxid was found to be unreliable, owing to the precipitation of appreciable amounts of silica that was dissolved by the mixed carbonates from the porcelain crucible. Determine the copper and lead, which remain as insoluble sulphids after the fusion, and the zinc, which remains in the original filtrate, according to the scheme described under the following method.

(b) MUNSON'S METHOD.

Treat 100 grams of the moist sample after evaporation to dryness, or 25 grams of the dry sample in a four-inch porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. Usually from 10 to 15 cc are sufficient for this purpose. Gently heat over a Bunsen burner until all danger of foaming is past, which will require not more than three minutes; then transfer the dish to a muffle furnace and keep it at a low red heat until all organic matter is destroyed. It is occasionally found necessary to add a few drops of nitric acid to completely destroy organic matter. When the material is completely ashed, allow the dish to cool; add 25 cc of hydrochloric acid (1 to 8) and evaporate on a water bath to dryness; take up with water and acidify with two or three drops of hydrochloric acid. Transfer to a beaker without filtering and treat with hydrogen sulphid. After heating upon a water bath for a few minutes the precipitate and the insoluble residue are collected upon a filter. The precipitate and residue may contain sulphids of tin, lead, and copper, and oxid of tin; the filtrate will contain any zinc that is present. Fuse the sulphid precipitate and insoluble ash residue with about three grams of caustic soda in a silver crucible for a half hour to render soluble any insoluble tin compounds. Dissolve the mass with hot water and slightly acidify with hydrochloric acid. Again treat with hydrogen sulphid without filtering. By this treatment all the tin is thrown down as sulphid with the sulphids of copper and lead. Collect the precipitate upon a filter and wash thoroughly with hot water. The filtrate may be rejected. To separate the tin sulphid from those of copper and lead, wash several times upon the filter with separate portions of 10 cc of strong boiling ammonium sulphid. Usually 50 cc of the ammonium sulphid will be found sufficient to completely dissolve all tin sulphid; but portions of the filtrate should be tested to make sure of this point. The filtrate is then made acid with hydrochloric acid to precipitate the tin sulphid, which, after standing for a few moments, is collected upon an ashless filter, ignited, and weighed as stannic oxid.

Treat the insoluble residue remaining from the ammonium sulphid washing with nitric acid, filter, wash, nearly neutralize with ammonia the excess of mineral acid, and add ammonium acetate, as there is usually a small amount of iron present. If any iron salt precipitates, filter, wash and divide the filtrate for the determinations of copper and lead. In the absence of lead, copper may be determined electrolytically, or it may be titrated with potassium cyanid. Unless added as a coloring agent, copper will seldom be present in sufficient quantity to warrant its determination.

^a Sutton, Volumetric Analysis, 8th ed., p. 373.

Precipitate lead with potassium chromate in an acetic acid solution; and weigh upon a tared filter as lead chromate.

Evaporate the filtrate from the hydrogen sulphid precipitate to about 60 cc; add bromin water to oxidize the iron salts, and any remaining hydrogen sulphid. Boil off the excess of bromin and, unless the solution is distinctly yellow, add a few drops of concentrated solution of ferric chlorid to make it so. Nearly neutralize the mineral acid with ammonia, and add ammonium acetate to precipitate iron phosphate and excess of iron. Filter and thoroughly wash the precipitate. To the filtrate, made distinctly acid with acetic acid and boiled, add hydrogen sulphid to precipitate zinc. Unless the zinc sulphid comes down white, it should be dissolved, again treated with ammonium acetate to remove traces of iron, and re-precipitated as sulphid. Finally collect the zinc sulphid upon an ashless filter, ignite and weigh as zinc oxid.

11.—“SOAKED” VEGETABLES.

A class of canned vegetables commercially known as “soaked” goods is now very commonly found upon the market, and constitutes the cheapest grade of vegetables sold. So far as the writer’s experience goes, only peas, beans, and corn, or combinations of these three, are found in this class. The material used for “soaked” products are the ordinary matured peas and beans, such as are used for seed, or are sold dried upon the market, and corn that has passed the stage when it can be supplied for the green market. The particular advantage in canning these goods is that the season for green vegetables passes rapidly, and in case the supply is greater than the canneries can handle, recourse is made to the packing of the matured product. Besides, these dried materials may be kept for some time, and thus serve to keep the canneries in operation during the less busy season.

So far as the composition of this class of canned vegetables is concerned, it probably varies but little from that of the younger vegetables, yet it does not possess the value as a relish that the former has. In the mature vegetables the percentage of total solids is much higher than in the young and more succulent vegetables, and this condition holds in the canned goods if only the solid contents of the can are considered. However, in a large number of samples of “soaked” goods examined, the proportion of liquid to solid portion was exceedingly high; so that when the entire contents of the can were taken the per cent of total solids was about normal for the green vegetables.

The detection of “soaked” vegetables is not a difficult matter for one who has had experience with this class of goods, but for a layman the task may not be so easy. As stated above, the high percentage of solids in the solid portion of the can is characteristic. Soaked peas and beans lose much, if not all, of their green color, and have the general appearance of the well-matured product. Their cotyledons are well formed, firm and mealy. With the pea the caulicle is particularly prominent, the process of soaking having been sufficient to start its development. With corn, the kernel is plump and hard and lacking in milky consistency. The succulence so characteristic of the green pea, bean, and corn is entirely lacking. The sense of taste may also be applied in the detection of this class of goods. From their nature it is difficult to apply specific tests, but a little practice will enable the analyst to detect them with reasonable certainty.

VIII.—COCOA AND ITS PREPARATIONS.

By F. T. HARRISON,

District Analyst, London, Ontario.

It has been found impossible to prepare the report on this subject this year. The heading is inserted here to preserve its proper order.

IX.—TEA AND COFFEE.

By W. H. ELLIS,
District Analyst, Toronto, Canada.

It has been found impossible to prepare the report on this subject this year. The heading is inserted here to preserve its proper order.

X. SPICES.

By A. L. WINTON,
Chemist of State Experiment Station, New Haven, Conn.

1.—GENERAL DISCUSSION.

The microscope is a most valuable means of detecting adulterants of vegetable origin in spices, as it usually discloses the particular adulterant present, even when in small amount.

Quantitative determinations are made, either to corroborate the results of the microscopical examination, or to detect exhausted spices, mineral matter, and other adulterants which do not have distinctive microscopic characters.^a

2.—PREPARATION OF SAMPLES.

Grind the sample so as to pass a sieve with round holes one millimeter in diameter. For the determination of starch in pepper by the diastase method, reduce a portion of the sample to an impalpable powder, by grinding in a mortar.

3.—DETERMINATION OF WATER.^a

Dry two grams at 110° C. to constant weight. From the loss in weight thus sustained subtract the amount of volatile ether extract determined as below described. This method, described by Richardson,^b gives a close approximation to the true percentage of moisture.

4.—DETERMINATION OF TOTAL ASH.

Follow the method of the Association.^c If calcium carbonate is present, care must be taken to burn the material and also the residue after exhaustion with water, at a heat below redness, thus avoiding loss of carbonic acid of the carbonate. When leaching with water is necessary, it is advisable to add a few drops of ammonium carbonate solution before evaporation.

5.—DETERMINATION OF ASH SOLUBLE IN WATER.

Boil the ash prepared as above with 50 cc of water, collect the insoluble portion in a Gooch crucible, wash with hot water, dry, ignite, and weigh.^d Subtract the percentage of insoluble ash thus determined from the percentage of total ash, thus obtaining the percentage of water-soluble ash.

6.—DETERMINATION OF "SAND" OR ASH INSOLUBLE IN HYDROCHLORIC ACID.

Incinerate 2 grams of the material as above directed, boil with 25 cc of 10 per cent hydrochloric acid (sp. gr. 1.050) for 5 minutes, collect the insoluble matter in a Gooch crucible, wash with hot water, ignite, and weigh.

^a See also Appendix, p. 152.

^b U. S. Dept. Agr., Div. Chem., Bul. 13, Part 2, p. 165.

^c U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 23.

^d Conn. Agr. Expt. Sta. Rept. 1898, p. 186.

7.—DETERMINATION OF LIME.

Calcium sulphate and carbonate are frequently used as adulterants, and are also present in appreciable amount in limed nutmegs, ginger, etc. In the presence of calcium sulphate, the water solution gives tests for both lime and sulphuric acid. Samples containing a considerable amount of carbonate effervesce on addition of 10 per cent hydrochloric acid. Determine lime in the ash, after separation of iron and alumina phosphates, as described under Baking Powder (p. 106.)

8.—DETERMINATION OF TOTAL SULPHUR.^a

(For mustard and samples adulterated with calcium sulphate.)

Convert about 10 grams of sodium peroxid into hydroxid in a nickel crucible by adding a little water and boiling over a low flame until the excess of water is expelled. Stir 1 gram of the material into the slightly cooled hydroxid and oxidize by gradually raising the heat and adding small portions of sodium peroxid until the oxidation is complete. Dissolve the fused mass in 400 cc of water, acidify strongly with hydrochloric acid, boil until the excess of peroxid is destroyed and chlorin expelled, filter through pure paper, make neutral with ammonia, and add an excess of 4 cc of concentrated hydrochloric acid. From the boiling solution precipitate sulphuric acid by gradually adding a solution containing 1 gram of barium chlorid. After standing over night, in a warm place, filter the barium sulphate, wash, ignite, and weigh.

Osborne has found commercial sodium peroxid to be freer from sulphur than most preparations of so-called chemically pure sodium hydroxid made from the metal, and as the former is very much cheaper than the latter, it is advantageous to use it as here described.

9.—DETERMINATION OF CARBON DIOXID.

(For samples adulterated with calcium carbonate.)

Proceed as directed under total carbon dioxid in Baking Powder (p. 98 and following).

10.—DETERMINATION OF VOLATILE AND NONVOLATILE ETHER EXTRACT.^b

Extract 2 grams of the ground material for 20 hours, in a continuous extraction apparatus, with absolute ether.^c Transfer the ethereal solution to a tared capsule and allow to evaporate at room temperature. Let stand 18 hours over sulphuric acid and weigh the total ether extract. Heat the extract gradually to 100° C., continue the heating at that temperature for 6 hours, and then at 110°, until the weight becomes constant. The loss is volatile oil; the residue, nonvolatile ether extract.

11.—DETERMINATION OF ALCOHOL EXTRACT.^d

Place 2 grams of material in a 100-cc flask and fill to the mark with 95 per cent alcohol by volume (sp. gr. 0.815 at 15.5° C.). Stopper, shake at intervals of 30 minutes for 8 hours, and allow to stand 16 additional hours without shaking. Filter the extract through a dry filter, evaporate 50 cc to dryness in a flat-bottomed dish on a water bath, and heat to constant weight at 110° C. The result is practically the same when the time of extraction is 48 instead of 24 hours. Winton, Ogden, and Mitchell,^e who describe this method, do not claim that it extracts all matter soluble in alcohol; in fact, the residue separated from the solutions by filtration, when

^a Osborne, Conn. Agr. Expt. Sta. Rept., 1900, p. 445.

^b Richardson, U. S. Dept. of Agr., Div. of Chem., Bul. 13, Part 2, p. 165.

^c See Appendix, pp. 153 and 154.

^d See Appendix, p. 154.

^e Conn. Agr. Expt. Sta. Rept., 1898, p. 187.

treated for 24 additional hours with a fresh portion of alcohol, yielded, in their experience, small additional amounts of extract. The method, however, gives nearly the full amount of extract and the results are concordant; whereas, extraction in a Soxhlet apparatus, if continued until no more extract is removed, is an interminable operation, and, as it is difficult to keep the strength and temperature of the extracting alcohol constant, gives results far from satisfactory.

12.—DETERMINATION OF COPPER-REDUCING MATTERS BY DIRECT INVERSION.^a

Extract 4 grams of the material on a Schleicher and Schuell's No. 589 blue-ribbon washed filter, or some other filter that will completely retain the smallest starch granules, with five successive portions of 10 cc of ether. After the ether has evaporated, wash with 150 cc of 10 per cent (by volume) alcohol. Weak alcohol is employed instead of water, because, as pointed out by Lindsey, it is not so liable to carry starch granules through the paper.

Since it is not possible to wash samples of Batavia cassia with water or dilute alcohol, owing to the formation of a glutinous mass which clogs the filter, for the sake of uniformity, all preliminary washing is best omitted in determinations made on all varieties of cassia, as well as on cassia buds and cinnamon.

Carefully wash the residue from the paper into a 500-cc flask, with 200 cc of water, using a small wash bottle, and gently rubbing the paper with the tip of the finger.

Convert the starch into dextrose by the Sachsse method,^b as follows:

Add 20 cc of 25 per cent hydrochloric acid (sp. gr. 1.125) and heat for three hours on a boiling water bath. Cool the solution nearly, but not quite, neutralize with sodium hydroxid solution, make up to 500 cc, and filter through a dry paper.

Determine reducing matters by the Allihn method,^c as follows:

Mix 30 cc of a solution containing 173 grams of Rochelle salts and 125 grams of caustic potash in 500 cc of water, and 30 cc of a solution of 34.69 grams of pure crystallized copper sulphate in 500 cc of water, in a beaker of 200 cc capacity and heat to boiling. To the boiling liquid, without delay, add 25 cc of the solution to be examined, and continue the heating until boiling begins again. After the reduced copper suboxid has settled, collect on a Gooch crucible, dry at a moderate heat, and finally heat for three to five minutes at dull redness, taking care to avoid a bright red heat and to allow access of sufficient air to complete the oxidation to copper oxid (after Bartlett^d). After weighing, repeat the heating to make certain that the oxidation is complete.

From the weight of copper oxid calculate the weight of metallic copper, using the factor 0.7986, and find the corresponding amount of dextrose in Table VIII. To obtain the corresponding weight of starch, multiply the weight of dextrose by 0.9.

If desired, the copper may be weighed as Cu_2O after washing with alcohol and drying at 100° C., or it may be determined electrolytically by one of the official methods.

To prepare asbestos pulp for use in the Gooch crucible, cut woolly asbestos (best quality) into small pieces, boil with hydrochloric acid, and wash free from acid and fine particles on a sieve with one-mm meshes. Woolly asbestos of suitable quality, when packed in the crucibles with the aid of a blunt glass rod, retains completely the finely divided copper suboxid, which is not true of the variety usually employed in filtering coarser precipitates.

Copper-reducing matters by direct inversion was first determined in pepper by

^a U. S. Dept. of Agr., Div. of Chem., Bul. 13: p. 166. Conn. Agr. Expt. Sta. Rept., 1898, p. 187. See also Appendix, p. 154.

^b Chem. Centralbl. 1877, 8, 732.

^c Jour. prakt. Chem., 1880, N. F., 22, 52.

^d Maine Agr. Expt. Sta. Rept., 1888, p. 207.

Lenz.^a Although useful, the results are not of as great value as those by the diastase method.

13.—DETERMINATION OF STARCH BY DIASTASE METHOD.^b

Extract 4 grams of the finely pulverized material with ether and 10 per cent alcohol, as described in the preceding section. Carefully wash the wet residue from the paper into a beaker with 100 cc of water, heat on an asbestos plate to boiling with constant stirring, and continue the boiling and stirring thirty minutes. Replace the water lost by evaporation, and immerse the beaker in a water bath kept at from 55 to 60°. When the liquid has cooled to the temperature of the bath, add 10 cc of fresh extract of malt (prepared by digesting for two or three hours 100 grams of powdered fresh malt with 1,000 cc of water and filtering), and digest the mixture for one hour, with occasional stirring. Boil a second time for fifteen minutes, cool, and digest as before with another 10-cc portion of malt extract. Heat to boiling the third time, cool, and make up the liquid to 250 cc in a graduated flask, filter through a dry paper, and remove 200 cc of the filtrate to a 500-cc flask. Conduct the inversion with acid, and determine the reducing power of the solution, as already described under "Copper-reducing matters by direct inversion," making a correction for the copper reduced by the added malt extract, as determined by blank analyses. The residue after the malt digestion, when examined microscopically, must be entirely free from starch.

Results by Winton, Ogden, and Mitchell^c show that cayenne pepper, mustard, and certain other materials, which are practically free from starch, yield very little or no copper-reducing matter, when treated by the method just described. This treatment is, therefore, without effect on the cellulose, pentosans, or other matters in the spices named, although they yield copper-reducing material on treatment with acid.

On the other hand, in decorticated white pepper and Jamaica ginger, which contain little besides starch that is affected by acid, practically the same results are obtained by the diastase method as by direct inversion with acids.

This determination of starch is very valuable as a means of detecting starchy adulterants in spices normally free from starch and nonstarchy adulterants in spices which contain starch.

14.—DETERMINATION OF CRUDE FIBER.^d

The method is that adopted by the Association of Official Agricultural Chemists for the analysis of cattle foods, except that the fiber is filtered and weighed on a paper rather than on a Gooch crucible, since the latter is liable to clog, rendering filtration impossible. Place the residue from the determination of ether extract in a 500-cc Erlenmeyer flask, and add 200 cc of boiling 1.25 per cent sulphuric acid. Loosely cover the flask, heat at once to gentle boiling, and continue the boiling thirty minutes. Filter on a paper, wash with hot water, and rinse back into the same flask with 200 cc of boiling 1.25 per cent sodium hydroxid solution, nearly free from carbonate. After boiling, as before, for thirty minutes, collect the fiber on a weighed paper, thoroughly wash with hot water, and finally with a little alcohol and ether. Dry to constant weight at 100° C., and weigh. Deduct the amount of ash in the fiber, as determined by incineration, from the total weight.

Determine the loss in weight sustained by the paper on treatment with sodium-hydroxid solution, alcohol and ether, and introduce the necessary correction.

^a Ztschr. anal. Chem., 1884, 23, 501.

^b Maereker, Handbuch der Spiritusfabrikation, 7th ed., 1898, p. 109; Wiley, Principles and Practice of Agricultural Analysis, 1898, Vol. III, p. 198.

^c Conn. Agr. Expt. Sta. Rept., 1898, p. 189.

^d See Appendix, pp. 154 and 155.

15.—DETERMINATION OF NITROGEN.

(a) KJELDAHL METHOD.

*(For all spices except black and white pepper.)*See methods of the Association of Official Agricultural Chemists.^a

(b) GUNNING-ARNOLD METHOD.

(For black and white pepper.)

Owing to the presence of piperine, the Gunning-Arnold method^b must be used to determine nitrogen in both black and white pepper. Mix 1 gram of the material in a 600-cc Jena^c flask with 1 gram of copper sulphate, 1 gram of mercuric oxid, 15 to 18 grams of potassium sulphate, and 25 cc of sulphuric acid. After heating gently until frothing ceases, boil the mixture from two to four hours. When nearly cool, add about 300 cc of water, 50 cc of potassium sulphid solution (40:1,000), and sodium hydroxid solution to alkaline reaction. Distill into standard acid and titrate with standard alkali, as usual.

16.—DETERMINATION OF NITROGEN IN NON-VOLATILE ETHER EXTRACT.—

WINTON, OGDEN, AND MITCHELL METHOD.

(For black and white pepper.)

Extract 10 grams of pepper for twenty hours in a continuous extraction apparatus with absolute ether, collecting the extract in a weighed Jena flask of about 250-cc capacity. Evaporate the ether, dry first at 100° C., and finally to constant weight at 110° C. Determine the nitrogen in the weighed extract by the Gunning-Arnold method, digesting in the same flask used for the extraction. Calculate the parts of nitrogen in 100 parts of the non-volatile ether extract.

Results on authenticated samples show that 100 parts of non-volatile ether extract from black pepper, owing to the presence of piperin, contain not less than 3.25 parts, and from white pepper not less than 4.00 parts of nitrogen. Linseed meal and other oily adulterants may contain about the same amount of ether extract as pepper, but this extract is practically free from nitrogen.

If desired, crude piperine may be calculated from the nitrogen by multiplying by 20.36.

17.—DETERMINATION OF COLD WATER EXTRACT.

(For ginger.)

Place 4 grams of ginger in a graduated 200-cc flask, add water to the mark, shake at half-hour intervals during 8 hours and let stand 16 additional hours, without shaking. Filter and evaporate 50 cc to dryness in a flat-bottomed metal dish. Dry to constant weight at 100° C.

Cold water extract was first determined by Allen and Moor^d as a means of detecting exhausted ginger. The process of Winton, Ogden, and Mitchell^e here described is more easily carried out and gives more concordant and somewhat higher results than extraction on a filter with the same volume of water added in consecutive portions. Complete extraction on a filter was found impracticable.

^a U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 14.^b Ztschr. anal. Chem. 1892, 31, 525; Conn. Agr. Expt. Sta. Rept., 1898, p. 190.^c See Appendix, p. 155.^d Analyst, 1894, 19, 124.^e Conn. Agr. Expt. Sta., Rept. 1898, p. 190.

18.—DETERMINATION OF TANNIN EQUIVALENT BY THE LÖWENTHAL-RICHARDSON METHOD.^a*(For cloves and allspice.)*

Extract 2 grams of material 20 hours with absolute ether. Boil the residue 2 hours with 300 cc of water, cool, make up to 500 cc and filter. Measure 25 cc of this infusion into a flask of about 1,200-cc capacity, add 20 cc of indigo solution and 750 cc of distilled water. Run in standard permanganate solution at the rate of one or two drops a second, with constant shaking, until a bright golden yellow color appears.

Determine in the same manner the number of cubic centimeters of permanganate solution consumed by 20 cc of indigo solution alone and subtract from the number consumed by the spice infusion and indigo solution together.

Indigo solution must be made from sodium sulphindigotate of best quality (such as is furnished by Gehe & Co., Dresden, or Grueber & Co., Leipzig), otherwise the titration will not be sharp. Dissolve 6 grams of the salt in 500 cc of water, with the aid of heat. Cool, mix with 50 cc of concentrated sulphuric acid, make up to 1 liter and filter.

Prepare standard potassium permanganate solution by dissolving 1.333 grams of the pure salt in 1,000 cc of water. Standardize by titration of 10-cc portions of decinormal oxalic acid solution (6.3 grams of the pure crystallized acid in 1,000 cc), which have been previously diluted to 500 cc, heated to 60° C. and mixed with 20 cc of dilute sulphuric acid (1:3 by volume). Add the permanganate solution slowly, with constant stirring, until a pink color appears. Ten cubic centimeters of decinormal oxalic acid solution are equivalent to 0.06232 gram of quercitannic acid, or 0.008 gram of "oxygen absorbed."

Ellis first recommended the determination of tannin as a means of detecting adulteration, but Richardson found that the abbreviated method here described is quite as useful as the more tedious process for the actual determination of tannin.

19.—MICROSCOPICAL EXAMINATION.

As already stated, the microscope is the most valuable means of detecting adulterants of vegetable origin in spices, as it usually discloses the particular adulterant present, even when in small amount. The analyst who undertakes this work should have a general knowledge of vegetable histology and microscopic manipulation, and should be thoroughly familiar with the microscopic appearance of the spices and the spice adulterants. He should have at his command the standard works on these subjects, and also a set of standard samples of all the materials likely to be encountered. The following works are especially recommended:

Moeller. Mikroskopie der Nahrungs- und Genussmittel aus dem Pflanzenreiche. Berlin, 1886.

Vogl. Die wichtigsten vegetabilischen Nahrungs- und Genussmittel. Berlin, 1899.

Tschirch und Oesterle. Anatomischer Atlas der Pharmakognosie und Nahrungsmittelkunde. Leipzig, 1900.

Only a few general hints are here given:

(a) APPARATUS.

Dissecting microscope or hand lens.

Compound microscope provided with $\frac{2}{3}$ and $\frac{1}{8}$ -inch objective, 1 and 2-inch oculars, double nosepiece, eyepiece micrometer, and polarizing apparatus.

A series of sieves with meshes ranging from 0.2 to 2 mm.

Slides, cover glasses, needles, scalpels, forceps, etc.

^a Allen. Commercial Organic Analysis, 1889, Vol. III, Part I, p. 109. U. S. Dept. of Agr., Div. of Chem., Bul. 13, Part 2, p. 167.

(b) MICRO-REAGENTS.

Of the numerous reagents employed in histological work, the following are most useful in spice examination:

Distilled water.

Glycerin, pure and diluted with equal volume of water.

Absolute alcohol.

Ether.

Ammonium hydroxid.

Potassium hydroxid (5 per cent).

Chloral hydrate (8 parts in 5 parts of water).

Schultze's macerating mixture: Crystallized potassium chlorate mixed with nitric acid as needed.

Iodin in potassium iodid: 0.05 gram iodine, 0.2 gram potassium iodid, and 15 cc water.

Chlorzinc iodine: Treat an excess of zinc with hydrochloric acid, evaporate to a thick sirup and filter through asbestos. As needed, saturate a small portion of the solution first with potassium iodid and finally with iodine.

Millon's reagent: Dissolve metallic mercury in its weight of concentrated nitric acid, add an equal volume of water, and decant off the clear liquid as soon as the precipitate settles.

Ferric acetate or chlorid solution.

Alkanna tincture, diluted with an equal bulk of water.

Aqueous solution of safranin.

Hydrochloric acid (10 per cent).

Acetic acid.

(c) PREPARATION OF THE MATERIAL.

Reduce a portion of the sample to a fine powder in a mortar. Separate another portion into several grades of fineness in sieves of different mesh or by jarring on a sheet of paper. In the coarser grades fragments of a suspicious nature may often be seen with the naked eye or under a simple microscope, which should be picked out with forceps for subsequent examination under the compound microscope.

(d) EXAMINATION.

Mount a small amount of the ground sample in water and examine under the compound microscope with both ordinary and polarized light. This gives a general insight into the nature of the material and serves for the detection and identification of starch granules and various tissues.

Draw a small drop of iodine solution into the same preparation by means of a piece of filter paper placed on the opposite edge of the cover glass and examine. Starch granules will be colored blue or blue-black, cellulose yellow, and proteids either brown or yellow.

In the manner described draw a little potassium hydroxid solution under the cover glass and examine once again. This treatment gelatinizes the starch granules, dissolves the proteids, saponifies the fats, and in other ways clears the preparation. It also imparts to tannins a reddish color.

If treatment with potash does not clear the tissues satisfactorily, treat a fresh portion for some hours with chloral hydrate solution.

Examine also the crude fiber obtained in the chemical analysis, as in this material the stone cells and other tissues are beautifully distinct.^a

To isolate stone cells, bast fibers, and other thick-walled cells macerate a portion of the sample in Schultze's liquid, using such proportion of potassium chlorate and

^aWinton, Conn. Agr. Expt. Sta. Rept., 1896, p. 34.

nitric acid and heating for such a time as secures the desired results. Powdered charcoal and charred shells resist the bleaching action of potash, chloral hydrate, and Schultze's liquid, the fragments after, as before the treatment, being black and opaque.

If it is desired to distinguish cellulose from infiltrated substances (lignin, suberin, etc.), add to a water mount freshly prepared chlorzinc iodine, which colors the former blue and the latter yellow.

As a test for proteids, cautiously warm, on a slide, with a drop of freshly prepared Millon's reagent. The proteids are partially disorganized, taking on gradually a brick-red color. If it is desirable to study the form of the aleurone (proteid) granules, which in some plants are quite as characteristic as starch granules, prepare a mount in pure glycerin or oil.

To distinguish fats, oils, essential oils, and resins from other cell contents, treat for an hour with alkanna tincture diluted with an equal bulk of water, which imparts to these substances a deep red color; or treat with ether, which dissolves them. Treat also with alcohol, which dissolves the essential oils and resins but does not perceptibly affect the fats and oils.

In testing for tannins and tissues impregnated with those substances, add ferric acetate or chlorid solution. Both of these reagents give with tannins a green or blue color, but the former acts more slowly and is to be preferred.

Crystals of calcium oxalate are recognized by their characteristic forms and their deportment with polarized light. To distinguish calcium oxalate from calcium carbonate, treat with acetic acid, which does not affect the former but dissolves the latter with effervescence. Both are soluble in hydrochloric acid.

Other special reagents may be occasionally useful, but as a rule, reagents play a subordinate part in the microscopic examination of spices, the chief factor being a thorough understanding of the size, shape, color, and other characteristics of the histological elements, which can be learned only by experience.

XI.—VINEGAR.

By WM. FREAR,

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Vinegars may be *defective* because of the poor quality of the liquids from which they are prepared or because of incomplete fermentation; *impure* from the development of abnormal fermentations, invasion of vinegar eels (*Anguillula oxophila*), moulds, or presence of foreign substances accidentally introduced, such as metallic salts formed by the action of the acid upon metallic vessels, faucets, etc.; *adulterated*, as by dilution, addition of mineral acids, pungent materials, coloring matters, etc.; *misbranded*, as when a pure vinegar of one sort is sold under the name of another. It is needful that methods for the detection of all the foregoing classes of variations shall be employed as occasion demands; that is, variation in normal constituents as well as presence of foreign matters must be detected and often measured.

1.—PREPARATION OF SAMPLES.

For microscopic examination, the original sample should be employed, but for chemical analysis the vinegar should be filtered, if turbid from the presence of suspended solids. Samples should be kept in glass bottles with ground-glass stoppers and should be promptly analyzed to avoid fermentative changes, which progress rapidly in the warm air of the laboratory.

2.—CALCULATION OF RESULTS.

Express all results as per cent by weight. Owing to the slight departure of the specific gravity of vinegars from that of water, no error of importance in most con-

trol work arises from considering the weight of one cubic centimeter of vinegar to be one gram. Where greater accuracy is desired, the quantities of vinegar employed may be accurately weighed, or the weight estimated from a direct determination of the specific gravity, or the average specific gravity of the kind of vinegar used may be taken as a means of a closer approximation than that attained by regarding the specific gravity of the vinegar to be 1.000. Blyth^a gives the following figures:

	Range.	Average.
Wine vinegar	1.0129-1.0213	1.0175
Spirit vinegar	1.008-1.013	1.0082
Malt vinegar		1.0150
Sugar vinegar		1.0100
Wood vinegar		1.0070

The writer has found in the case of cider vinegars a range of 0.997 to 1.029, with an average of 1.015.

3.—DETERMINATION OF SPECIFIC GRAVITY.

Proceed as directed under wines (p. 82).

4.—DETERMINATION OF TOTAL SOLIDS OR EXTRACT.

Evaporate 10 cc in a tared platinum dish of 50-mm diameter on the steam bath to a sirupy consistency, dry for 2½ hours in the drying oven at the temperature of boiling water, cool and weigh.

5.—DETERMINATION OF ASH.

Use the method employed in case of wines (p. 83).

6.—DETERMINATION OF SOLUBILITY, ALKALINITY, AND PHOSPHORIC ACID OF ASH.

Smith's method, modified by Frear:^b Evaporate 25 cc to dryness, burn, cool, weigh; extract the ash repeatedly with hot water on an ash-free filter; dry and ignite the filter with undissolved residue, cool and weigh, and calculate as insoluble ash. Titrate the aqueous extract with decinormal acid, using methyl orange as indicator. Acidulate the neutralized solution with nitric acid; also treat the insoluble ash with nitric acid, and precipitate the phosphoric acid from the two solutions separately by molybdate solution. The yellow precipitates, after solution in ammonia, may either be treated with magnesia mixture, as in ordinary ash analysis, or reduced with zinc and sulphuric acid, and titrated with standard permanganate solution. Express the results in terms of milligrams of P_2O_5 in the water-soluble and water-insoluble ash, respectively, from 100 grams of vinegar.

7.—DETERMINATION OF TOTAL ACIDITY.

Dilute 12 cc in a beaker until the solution appears very slightly colored when viewed against a white background, add a few drops of phenolphthalein indicator, and titrate with half-normal sodium hydroxid. Multiply the number of cubic centimeters of half-normal soda solution required by 0.03 for the total acidity expressed as grams of acetic acid. When 12 grams of vinegar are employed in the determination, the number of cubic centimeters of half-normal alkali employed may be divided by 4 for the percentage of total acids expressed in terms of acetic acid.

^a Foods: Their comp. and anal., 4th ed., p. 582.

^b Jour. Am. Chem. Soc., 1898, 20, 5.

8.—DETERMINATION OF VOLATILE AND FIXED ACIDS.

(a) VOLATILE ACIDS.

Heat 15 cc of the vinegar to boiling in a flask, adding a little tannin if foaming occurs; then lower the flame and pass a current of steam through the vinegar to a condenser. Continue the operation until 15 cc of distillate shows no acidity upon a test with sensitive litmus paper. Titrate the combined distillate with half-normal sodium hydroxid, using phenolphthalein as indicator. The number of cubic centimeters of alkali required, multiplied by 0.03, gives the weight of volatile acids, expressed as grams of acetic acid.

(b) FIXED ACIDS.

Deduct volatile acids from total acids and multiply the remainder by 0.817 for sulphuric acid, or 1.117 for malic acid. Or dilute the nonvolatile residue from the distillation with water until the solution appears nearly clear against a white background. Titrate with half-normal sodium hydroxid, using phenolphthalein as indicator, as in case of the volatile acids. The weight of fixed acids, calculated to sulphuric or malic, is calculated by the factors given above. When 15 cc are taken, multiply the number of cubic centimeters of half-normal alkali solution employed by 0.163 for the percentage of fixed acids expressed in terms of sulphuric acid (H_2SO_4), or by 0.223 to express in terms of malic acid.

9.—DETECTION OF FREE MINERAL ACIDS.

(a) FIRST METHOD. ^a

Prepare an extract of logwood by pouring 100 cc of boiling water upon 2 grams of fresh logwood chips, allowing the decoction to stand for a few hours and filtering. Separate drops are spotted on a porcelain surface and dried over a water or steam bath. Add to one of the spots a drop of the vinegar to be tested (after concentration, if thought desirable); again evaporate to dryness. A yellow tint remains if free mineral acids are absent, a red tint if present.

(b) SECOND METHOD.

To 5 cc of vinegar add 5 or 10 cc of water; after mixing well, add 4 or 5 drops of an aqueous solution of methyl-violet (one part of methyl-violet 2B in 10,000 parts of water). The occurrence of a blue or green color indicates the presence of a free mineral acid.

10.—DETERMINATION OF FREE MINERAL ACIDS.

(a) HILGER'S METHOD.

Neutralize 20 cc of the vinegar exactly with half normal alkali, the end reaction being determined by the action of drops of the liquid upon sensitive violet litmus paper. Evaporate the neutral liquid to one-tenth volume in a porcelain dish, add a few drops of methyl-violet solution (that mentioned in paragraph 9), dilute with 3 or 4 cc of water, if needful, to secure a clear solution, bring to boiling, and titrate with half normal sulphuric acid till a green or blue color begins to appear. The difference, in cubic centimeters, between the seminormal alkaline and acid reagents added, multiplied by the factor 0.1225, expresses the percentage of mineral acid present, in terms of sulphuric acid (H_2SO_4).

(b) HEHNER'S METHOD.

To a weighed quantity of the sample add excess of decinormal alkali, evaporate to dryness, incinerate and titrate the ash with decinormal acid. The difference between

^a Ashby, Allen's Com. Org. Anal. 2d ed., vol. I, p, 393.

the number of cubic centimeters of alkali added in the first place and the number of cubic centimeters needed to titrate the ash represents the equivalent of the free acid present.

11.—DETERMINATION OF OXALIC ACID.

The presence of oxalic acid may be detected and its quantity determined by addition of a solution of calcium sulphate to a measured quantity of the vinegar.

12.—DETERMINATION OF ALCOHOL.

Domestic fruit vinegars are often incompletely acetified; if the specific gravity be abnormally low, a determination of the alcohol present is desirable. Owing to the small quantities usually present, it is best to concentrate the distillates. Carefully neutralize 100 cc of the vinegar and distill over 40 cc; redistill the distillate until 20 cc has passed over; cool to 15.5° C. and make up to 20 cc with distilled water. Determine the specific gravity by means of a pycnometer and calculate the percentage by weight, by Table II. The percentage in the last distillate, divided by 5, represents the amount in the original vinegar.

13.—DETECTION OF COLORING MATTERS.

The principal coloring matter used for tinting imitation vinegars in America is caramel. To detect this use Amthor's method (p. 120). A further test of the caramel may be made by boiling the aqueous solution of a portion of the precipitate obtained by Amthor's method, with Fehling's solution; caramel has a reducing action.

In the case of wine vinegars, test for foreign red colors may be made according to the methods given on p. 111 and following.

14.—DETECTION OF FOREIGN PUNGENT MATERIALS.

Exactly neutralize a portion of the vinegar (the residue from determination of total acidity may be used), evaporate off a portion of the water, and test the concentrated solution by taste for pungent substances; then agitate the liquid with ether, in a separatory funnel, remove and evaporate the ethereal layer, and apply the same test to the residual ethereal extract. The perfect identification of the specific substance employed is rarely attained.

15.—DETECTION AND DETERMINATION OF METALLIC POISONS.

Evaporate from 200 to 500 cc to dryness. In case of cider, malt, and other vinegars rich in solids, add a little sodium hydroxid and potassium nitrate, and incinerate. The ash thus obtained, or the solids themselves, in case of vinegars low in extract, is carefully dissolved in hydrochloric acid and the solution subjected to examination by methods indicated for the analysis of canned vegetables (p. 52).

16.—DETECTION OF PRESERVATIVES.

Preservatives are sometimes, though rarely, added to vinegar. Salicylic and benzoic acids may be separated by agitation with ether and detected in the residue left upon evaporation of the ether. Boric acid may be detected in the ash left upon the evaporation and incineration of the vinegar after neutralization by alkali.

Most of the tests for the detection of formaldehyde are excluded from use in testing vinegars, since the indicative reactions are usually produced by acetaldehyde also, which is frequently present in normal vinegar. From 100 cc of the vinegar distill 20 cc. To a portion of the distillate add a few drops of milk; float the liquid upon 90 to 94 per cent sulphuric acid to which a little ferric chlorid has previously been added. A violet ring appears at the point of separation of the liquids, if formaldehyde is present. Acetaldehyde does not produce this reaction, the color being yellowish green changing to brown.

A portion of the distillate prepared as above may, after the addition of a few drops of milk, be treated with a drop of concentrated aqueous ferric chlorid solution, agitated and well mixed with a nearly equal volume of concentrated hydrochloric acid. Warm below boiling with constant agitation. Shortly before ebullition a purple coloration of the casein appears, if formaldehyde be present; it is not produced by acetaldehyde.

17.—DETERMINATION OF THE SOURCE OF A VINEGAR.

It is not always possible to make this distinction with entire certainty. The principal vinegar of the United States is cider vinegar, though malt vinegar finds preference with many. The substitutes are chiefly (a) low wine vinegar, either sold under the name "white wine vinegar" or colored by addition of caramel, or even given color and body by addition of cheap apple jelly; (b) vinegar from sugarhouse wastes; (c) wood vinegar, or preparations from vinegar essence, with or without coloring matters. Grape, or true "wine vinegar," is important in few localities outside of California. Glucose vinegar is sometimes found.

The nature of the vinegar is commonly indicated, if it be pure of its kind, by its flavor and odor. The fruity quality of cider vinegar is usually very conspicuous; the odor of malt vinegar is characteristic; and impure wood vinegar often shows a very perceptible empyreumatic quality. Even when these qualities are distinctly indicative of the source of the vinegar, additional evidence is desirable for legal proof; often slight impurities mask them.

The *quantity of the solids* is often distinctive. The range for the principal vinegars is: Cider vinegar, 1.18 to 8.04; average, about 2.5. Malt vinegar, 1.75 to 6.0; average, about 3.0. Spirit vinegar, 0.13 to 0.78; average, about 0.3. Wine vinegar, 1.38 to 3.19; average, 1.9.^a The quantity of solids in sugar and glucose vinegars varies with the conditions of manufacture, sometimes corresponding closely with that of fruit vinegars. That of wood vinegar resembles the quantity in spirit vinegars. By the addition of foreign solids to spirit or wood vinegar the value of this criterion is often destroyed.

The *quality of the vinegar solids*, as a whole, is usually characteristic. The *consistency* of that from cider is thick and viscid or mucilaginous; that from sugar, glucose, or malt is somewhat more glutinous. The *odor* of baked apples is notable in cider-vinegar solids, that of molasses is often apparent in sugar-house vinegar, and that of malt vinegar is usually distinctive. The *flavor* of cider-vinegar solids is acid and somewhat astringent; in these respects wine vinegar resembles it. The bitter taste of caramel is usually observed in sugar-house vinegar solids and in those from colored spirit and wood vinegars. On burning the solids, the apple odor is developed by cider vinegar, that of burnt sugar by sugar-house vinegar, and that of scorched corn^b by glucose vinegar. The *solubility of the solids in alcohol* marks fruit vinegars—except a granular residue of tartar in grape vinegar—while the solids of malt and glucose vinegars are only very slightly dissolved.^c By addition of cheap cider jelly to spirit or wood vinegar, the characteristic apple quality is, however, given to the vinegar solids.^d

The *quantity of the ash* is useful in distinguishing spirit and wood vinegars from fruit and malt vinegars, the quantity in the former case rarely exceeding 0.1 per cent; in the latter rarely falling below 0.2; the range for pure cider vinegar is 0.19 to 0.57; average about 0.35 per cent.

The *quality of the ash* is far more indicative. That of fruit vinegars and malt vinegars is distinctly alkaline; that of spirit and wood vinegars very slightly so. The

^a According to Blyth. Eckenroth gives 0.35 to 1.51 per cent.

^b Davenport, 26th Ann. Rept. Milk Inspector, City of Boston, 1885.

^c Allen, Com. Organic Anal., Vol. I, p. 389.

^d Frear, Rept. Pa. Dept. of Agr., 1898, p. 138; Leach, Rept. Mass. State Bd. of Health, 1898, p. 633.

alkalinity^a expressed in terms indicated in the previously described methods is, for cider vinegar 26 to 65, average 39; for malt vinegar 5.5, and for spirit vinegar 1.1. The quantity of phosphoric acid in the ash, expressed in milligrams per 100 grams of the vinegar, ranges in case of pure cider vinegar from 9 to 39; in malt vinegar it is variously stated to range from 9 to 12.5 and from 50 to 100; while the ash of spirit vinegar shows traces only. The solubility of the phosphoric acid in cider vinegar is high, 50 to 75 per cent of the amount present being soluble in water, or from 4.7 to 22.7 mg per 100 grams of vinegar; no soluble phosphoric acid is found in the ash of spirit vinegar. If cider vinegars be diluted with hard water, the relatively insoluble earthy phosphates are formed and the proportion of soluble phosphoric acid greatly reduced.

The flame reaction obtained by igniting a drop of the solids upon a loop of platinum wire in a colorless flame is, in case of cider vinegar, exclusively that of potash; solids obtained from artificially colored vinegars, sugar, spirit, and glucose vinegars always give the sodium flame.^b

The optical activity of vinegars is often characteristic. For this purpose they should be examined in a 400-mm tube, after treatment with lead subacetate or boneblack, if necessary, for their clarification. Pure cider vinegar is slightly levorotatory, ranging from 0.96° to 2.79° Ventzke, in thoroughly acetified samples. Vinegar containing unfermented apple solids will be much more highly levorotatory; wine vinegar is also slightly levorotatory. On the other hand, vinegar from sugarhouse wastes is dextrorotatory before and levorotatory after inversion; that from glucose dextrorotatory both before and after inversion.^c Incompletely acetified vinegar made from incompletely fermented cider might exhibit the optical properties of a mixture of spirit vinegar and cider jelly, but the latter would be distinguished by differences at other points.

The ratio of reducing sugars, after inversion, to total solids is a means of distinguishing genuine cider vinegar from spirit vinegar supplied with solids from cider jelly. Where they constitute 50 per cent or more of the solids, the vinegar may be regarded as not pure cider vinegar. An incompletely fermented cider which had begun acetification might be mistaken for the imitation product if this ratio alone were considered; but low acetic acid, high alcohol, and other characters, make it easy to distinguish the former. The determination should be made as follows: Pipette 25 cc of the vinegar into a 100-cc flask; add 5 cc of concentrated hydrochloric acid; heat the flask in a water bath at 70° C., and after the vinegar has attained a temperature of 67°, maintain it at 67° to 70° for five minutes; then cool. Dilute one half; neutralize exactly with sodium hydroxid solution, using phenolphthalein as an indicator; make up to 100 cc and withdraw 25 cc for determination of reducing sugar by the copper-reduction method of Allihn, computing the results by the table of Meissl and Wein.^d In case a large ratio of reducing sugar to total solids be found, its origin—whether from cider jelly, sugarhouse wastes, or glucose—remains to be determined, either by collateral evidence or by a more detailed separation of the several sugars in the solids. For the latter purpose, employ the methods described by Browne.^e

The ratio of ash to solids is also indicative. In cider vinegar the ratio ranges from

^a Smith, A. W., Jour. Am. Chem. Soc., 1898, 20, 7; Blyth, Foods: Their Comp. and Anal., 4th ed., p. 587; Hehner, Vtschr. Chem., Nahr, 1893, 7, 194.

^b Davenport, 26th Ann. Rept. Milk Inspector, City of Boston, 1885.

^c Frear, Rept. Pa. of Dept. of Agr., 1898, p. 145; C. A. Browne, jr., Rept. Pa. State College Agr. Exp. Station, 1900, p. 265; also Bulletin 58, Pa. Dept. of Agr., p. 43; Doolittle & Hess, Jour. Am. Chem. Soc., 1900, 22, 19; Allen, Com'l Organic Analysis, second edition, Vol. I, p. 81; Von Bitteryst, Rev. fals. internat., 1894, 7, 151.

^d Wiley, Agricultural Analysis, Vol. III, p. 159.

^e Rept. Pa. State College Agr. Expt. Sta., 1900, p. 269-274; Cf. also, Frear, Rept. Pa. Dept. of Agr., 1898, p. 138 and following, and Browne, Bul. 58, Pa. Dept. of Agr., for comparative data.

4.6 to 17.1, average 9.0; that of pure spirit—malt and wine vinegar—averages 5 to 8; but upon addition of cider jelly to a vinegar, low in solids, the ratio becomes 17.1 to 80.

The *quantity of nitrogen* serves to distinguish malt vinegars from such as are derived from saccharine liquids, low wines, or wood acids. Calculated as albuminoids, the amount in malt vinegars is 0.65 to 0.7 per cent;^a in cider vinegars, 0.006 to 0.024 per cent;^b in sugar, glucose, spirit, and wood vinegars, much less.

The *presence of alcohol* in vinegars derived from alcoholic liquids often serves to distinguish them from wood vinegar; its absence is not conclusive against their derivation from the former class of materials.

The presence of *tartar* distinguishes wine vinegar, though its absence is not conclusive of other origin.^c Allen's method for this test is as follows:^d Treat the residue left from evaporation of the vinegar, with alcohol; a granular residue of tartar remains undissolved; to prove its character, pour off the alcohol and dissolve the residue in a small quantity of hot water. On cooling the aqueous solution, and stirring the sides of the vessel with a glass rod, the acid tartrate of potassium will be deposited in streaks on the track of the rod. An addition of an equal bulk of alcohol makes the reaction more delicate.

The *adulteration of wine vinegar* by addition of *free tartaric acid* is proved in a similar manner, the alcoholic solution of the extract is treated with an alcoholic solution of potassium acetate; upon stirring the mixture with a glass rod in a beaker, streaks and probably a distinct precipitate of tartar will be deposited. Quantitative results can be obtained upon titration of the precipitate by standard alkali.

The presence of *malic acid* distinguishes cider vinegars, though the quantity is often small. Failure to obtain a precipitate upon the addition of a few drops of neutral lead acetate to 10 cc of a vinegar, proves it not to be cider vinegar; if a precipitate be obtained, parallel tests with silver nitrate and barium chlorid to determine the absence of chlorids and sulphates should be made, before the presence of malic acid be considered proved.

Dextrin is often found in glucose vinegar and is precipitated from the concentrated vinegar upon addition of three or four volumes of strong alcohol; the precipitate may be identified by the physical properties and by its color reaction with iodine solution. Dextrin is also of general occurrence in malt vinegar.

Wood vinegar is quite commonly marked by the presence of *empyreumatic matters*; these are sometimes sufficient to impart their characteristic flavor to the vinegar. It has been recommended that the method of Cazeneuve and Cotton^e be used for their detection; this depends upon the immediate reduction of 1 cc of a 0.1 per cent solution of potassium permanganate when added to 10 cc of the liquid to be tested. Obviously this test is not applicable in the presence of caramel or the reducing sugars of fruit and malt vinegars; both the distillate and the ether extract of cider vinegars cause rapid reduction. The test is not applicable therefore to mixtures of wood vinegar with that from other sources, but may be useful in completing the examination of a vinegar shown by other evidence to be wood vinegar.

Microscopic examination may establish the absence of alcoholic and acetic ferments; in such event, the article is shown to be distilled vinegar.

^a Blyth, *Foods, Their Comp. and Anal.*, 4th edition, p. 587.

^b Frear, Rept. Pa. Dept. of Agr., 1898, p. 145.

^c Tretzol (*Forschungsber.*, 1896, **3**, 186; *Vjschr. Chem. Nahr.*, 1898, **11**, 257) states that true wine vinegars are occasionally found without tartar or more than traces of it. H. Eckenroth (*Pharm. Ztg.*, 1889, **34**, 14; *Vjschr. Chem. Nahr.*, 1891, **4**, 88) claims that it is always found, if from 500 cc to 1 liter of the vinegar be used for the test. Von Bitteryst (*Op. cit.*, 1896, **9**, 425) gives 0.04 gram per 100 cc as a minimum.

^d Commercial Organic Analysis, 2d ed., vol. 1, p. 389.

^e Vereinbarungen zur einheitlichen Untersuchung und Beurtheilung von Nahrungs- und Genussmitteln, II, 83. *Bul. soc. chim.*, 1881, [2], **35**, 102.

XII.—FLAVORING EXTRACTS.

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Flavoring extracts consist of three classes of preparations—tinctures, spirits, and artificial essences. Each class needs specific treatment, varying in detail with the object sought. In quantitative examination of either tincture of vanilla or spirits of lemon, it is essential to carry out comparative tests upon similar products of known purity and strength. Many of the methods recommended for lemon may be readily adapted to the examination of other spirits containing volatile oils.

(A) VANILLA AND ITS SUBSTITUTES.**1.—DETERMINATION OF TOTAL SOLIDS.**

Weigh 25 grams of the extract into a large, flat-bottomed dish which contains enough freshly ignited asbestos to absorb it; dry from twenty to twenty-four hours in a water-jacketed oven.

2.—DETERMINATION OF ASH.

Weigh about 10 grams of the extract in a flat-bottomed platinum dish, evaporate to dryness on a water bath, heat slowly until intumescence ceases, and ignite in a muffle at a low red heat until a white ash is obtained.

3.—EXAMINATION OF ASH.

Proceed as directed on page 106.

4.—DETECTION AND DETERMINATION OF COUMARIN AND VANILLIN.^a

Dealcoholize 50 grams of the extract in a glass evaporating dish upon a water bath at a temperature of about 80° C.; add water from time to time to retain the original volume. After removal of the alcohol, add normal lead acetate solution, drop by drop, until no more precipitate forms. Stir with a glass rod to facilitate flocculation of the precipitate. Filter through a moistened filter; wash three times with a few cubic centimeters of hot water. Cool the filtrate and extract with ether by shaking out in a separatory funnel. Use 15 to 20 cc of ether at each separation and repeat the shaking out three or four times, or until a few drops of the ether evaporated upon a watch glass leaves no residue. Place the combined ether extracts containing all of the vanillin and coumarin in a clean separatory funnel and shake out repeatedly with from 5 to 10 cc of 7 per cent ammonia.^b Repeat the treatment with ammonia once or twice after the fractions cease to be colored yellow.

Set aside the combined ammoniacal solutions for the determination of vanillin.

Wash the ether solution into an evaporating dish and allow the ether to evaporate at ordinary temperature. Extract the residue by treating it at room temperature with 5 to 10 cc of a petroleum fraction boiling between 30° and 40° C.; allow it to stand several minutes and then decant into a dried, weighed evaporating dish. Repeat the extraction with petroleum ether until a drop evaporated on a watch glass leaves no residue. Allow the petroleum ether to evaporate at room temperature; dry in a desiccator over sulphuric acid and weigh as coumarin.

The residue should be crystalline and have a melting point of 67° C. This, with the characteristic odor of coumarin, obtained by warming slightly, is sufficient for its identification.

^a Hess and Prescott, Jour. Am. Chem. Soc., 1899, 21, 256.

^b Winton prefers 2 per cent ammonia.

Slightly acidulate the ammoniacal solution reserved for vanillin with 10 per cent hydrochloric acid. Cool and shake out in a separatory funnel with 10 cc of chloroform, repeating the process as described for ether extraction. Evaporate the chloroform at room temperature and dry over sulphuric acid, or in an air bath at a temperature not exceeding 55°.

Wash the residue with boiling petroleum ether, using 5 to 10 cc at each treatment; decant the fractions into a dry, weighed evaporating dish. Evaporate the petroleum ether at room temperature and dry as before. Weigh the residue as vanillin.^a This should be pure and crystalline and have a melting point of 80° to 81°. This, with the characteristic odor and crystalline form, is sufficient for its identification as vanillin.

5.—DISTINCTION OF TRUE EXTRACT OF VANILLA FROM LIQUID PREPARATIONS OF VANILLIN.^b

(a) THEORY.

The leading fragrant principle of the vanilla bean and of true vanilla extract is vanillin, a definite chemical compound—hydroxymethoxybenzoic aldehyde. It is not the only fragrant or valuable constituent of vanilla bean and true vanilla extracts. The artificial vanillin, made for the market, contains vanillin identical with the vanillin contained in the vanilla bean; but the vanilla bean, as the vanilla extract, contains among its many "extractive matters" which enter into the food and fragrant value of vanilla extract, certain resins which can be identified with certainty in analysis by a number of determining reactions. Extract made without true vanilla can be detected by negative results in all these reactions.

Vanilla beans contain 4 to 11 per cent of this resin. It is of a dark red to brown color and furnishes about one-half the color of the extract of vanilla. This resin is soluble in 50 per cent alcohol, so that in extracts of high grade, where sufficient alcohol is used, all resin is kept in solution. In cheap extracts, where as little as 20 per cent of alcohol by volume is sometimes used, an alkali—usually potassium bicarbonate—is added to aid in getting resin, gums, etc., in solution, and to prevent subsequent turbidity. This treatment deepens the color very materially.

(b) METHOD OF ANALYSIS.

Place 50 cc of the extract to be examined in a glass evaporating dish and evaporate the alcohol on the water bath. When alcohol is removed, make up about the original volume with hot water. If alkali has not been used in the manufacture of the extract, the resin will appear as a flocculent red to brown residue. Acidify with acetic acid to free resin from bases, separating the whole of the resin and leaving a partly decolorized, clear supernatant liquid after standing a short time. Collect the resin on a filter, wash with water, and reserve the filtrate for further tests.

Place a portion of the filter with the attached resin in a few cubic centimeters of dilute caustic potash. The resin is dissolved to a deep red solution. Acidify. The resin is thereby precipitated.

Dissolve a portion of the resin in alcohol; to one fraction add a few drops of ferric chlorid; no striking coloration is produced. To another portion add hydrochloric acid; again there is little change in color. In alcoholic solution most resins give color reactions with ferric chlorid or hydrochloric acid.

To a portion of the filtrate obtained above add a few drops of basic lead acetate. The precipitate is so bulky as to almost solidify, due to the excessive amount of organic acids, gums, and other extractive matter. The filtrate from this precipitate is nearly, but not quite, colorless.

^aSee Appendix, p. 155.

^bJour. Am. Chem. Soc., 1899, 21, 719.

Test another portion of the filtrate from the resin for tannin with a solution of gelatin. Tannin is present in varying but small quantities. It should not be present in great excess.

6.—DETERMINATION OF CANE SUGAR.

See methods given on page 85.

7.—DETERMINATION OF ALCOHOL.

Proceed as directed on page 82.

8.—TESTS FOR COLORING MATTER—CARAMEL.

Of the artificial colors used in vanilla extracts, the most common is caramel.

Preliminary test.—If on shaking the bottle of vanilla the bubbles formed are of a bright caramel color, and they keep this color until the very last are gone, it indicates presence of caramel. This difference is readily shown by comparison with known pure samples.

Lead acetate test.—The coloring matter present in vanilla, or tonka, extracts is almost completely removed when the dealcoholized extract is treated with a few cubic centimeters of basic lead acetate solution. When caramel is present, the filtrate and precipitate, if any, have the characteristic red-brown color of caramel.

Phenylhydrazin hydrochlorid test.—To 20 cc of the extract add 1 cc of zinc chlorid (5 per cent solution), and then add 1 cc of a 2 per cent solution of potassium hydroxid and stir the precipitate with the solution. Filter and wash the precipitate with hot water. Dissolve the precipitate in 10 cc or more of a 10 per cent solution of hot acetic acid, receiving this solution in an evaporating dish; evaporate to 5 cc over water bath. Neutralize the excess of acetic acid with potassium hydroxid solution and divide the solution equally in two 6-inch test tubes. Add to each 5 to 10 cc of a solution of phenylhydrazin hydrochlorid and sodium acetate, prepared by dissolving 2 grams of phenylhydrazin and 3 grams of sodium acetate in 15 cc of water. Let one tube stand overnight. Heat the other on a water bath for half an hour. A brown precipitate will be obtained in both cases if caramel be present.

Fullers' earth may be substituted for zinc hydroxid. In such case, stir or shake a small amount of the earth with the extract, filter and wash with cold water. Extract the caramel with boiling water. Test the concentrated water solution with phenylhydrazin as above.

(B) LEMON EXTRACT.

These extracts are made by the solution of oil of lemon or its more soluble constituents in alcohol of varying strength. Oil of lemon contains nearly 90 per cent of terpenes, largely d-limonene, giving the oil a rotary power of 59 to 64 circular degrees at 20° F. The aromatic constituents are oxygenated bodies, the principal of which is citral. The latter bodies being more soluble in weak alcohol frequently constitute the chief flavoring substance present in pure extracts of the cheaper quality.

Extracts of the highest strength and purity are made by solution of the whole oil of lemon in deodorized alcohol, as described in the United States Pharmacopoeia. The lowest quality of adulterated extracts may contain minute amounts of citronella aldehyde or citral obtained from "lemon grass" (*Andropogon citratus*), together with aromatic or pungent tinctures, such as mace or capsicum.^a

1.—DETERMINATION OF TOTAL RESIDUE.

Evaporate 10 grams of the extract on a water bath at a temperature below the boiling point of the alcohol. In the absence of glycerol dry to a constant weight at

^a Dr. William Frear has suggested that if a few drops of bromin water are added to 3 cc of true lemon extract, the bromin will be almost instantaneously absorbed by the terpene present.

100° C. Examine the residue for sugar and glycerol. The presence of capsicum may be readily detected by taste.

2.—DETERMINATION OF GLYCEROL.

Proceed as directed on page 82.

3.—DETERMINATION OF ASH.

Ignite the residue from 10 grams of the extract at a dull red heat. Examine the ash for magnesia. Where the extract has been made with insufficient alcohol to effect complete solution of the oil, the liquid is frequently clarified by filtration with magnesia; in which case the latter substance may be detected in the ash.

4.—DETERMINATION OF SPECIFIC GRAVITY.

Determine the specific gravity as directed on page 82.

5.—DETERMINATION OF ALCOHOL.

(a) Dilute 50 cc to 200 cc; pour mixture into dry Erlenmeyer flask containing 5 grams of light carbonate of magnesia. Stopper, shake well, and filter quickly through a large, dry, plaited filter. Determine the alcohol in 150 cc of the filtrate, as directed on page 82, multiplying the results so obtained by $\frac{4}{3}$ to correct for the aliquot part taken.

(b) In the absence of appreciable quantities of solids or glycerol, calculate the alcohol approximately from the specific gravity of the extract, using Table II.

6.—DETECTION OF METHYL ALCOHOL.

These methods consist in the conversion of the alcohols to aldehydes by the method of Mulliken and Scudder,^a the removal of acetaldehyde, and the detection of formaldehyde when produced.

Dilute a portion of the distillate obtained in the determination of alcohol (or, for preliminary examination, dilute and filter the original extract) until the liquid contains approximately 12 per cent of alcohol by weight.

Oxidize 10 cc of the liquid in a test tube as follows: Wind copper wire 1 mm thick upon a rod or pencil 7 to 8 mm thick in such a manner as to inclose the fixed end of the wire and to form a close coil 3 to 3.5 cm long. Twist the two ends of the wire into a stem 20 cm long and bend the stem at right angles about 6 cm from the free end, or so that the coil may be plunged to the bottom of a test tube, preferably about 16 mm wide and 16 cm long. Heat the coil in the upper or oxidizing flame of a Bunsen burner to a red heat throughout. Plunge the heated coil to the bottom of the test tube containing the diluted alcohol. Withdraw the coil after a second's time and dip it in water. Repeat the operation from three to five times, or until the film of copper oxide ceases to be reduced. Cool the liquid in the test tube meanwhile by immersion in water. Remove 10 drops of the liquid so oxidized to a small porcelain capsule and reserve this for a separate test.

(a) REMOVAL OF THE ACETALDEHYDE BY PRESCOTT'S METHOD.^b

Add to the liquid remaining in the test tube 6 cc of a 3 per cent solution of hydrogen peroxid or an equivalent amount. Mix and filter into a porcelain dish. After three minutes add 2 cc of a 10 per cent solution of sodium thiosulphate. After two or three minutes place the dish in a good white light and test for formaldehyde.

Test for formaldehyde.—To the contents of the dish add 3 cc of a phloroglucin solu-

^aS. P. Mulliken and H. Scudder, Amer. Chem. Jour., 1899, 21, 266.

^bPharmaceutical Archives, 1901, vol. 4, No. 5.

tion made by dissolving 1 gram of phloroglucin and 20 grams of sodium hydroxid in sufficient water to make 100 cc. A bright red coloration (not purple) indicates the presence of methyl alcohol in the original sample. When too little hydrogen peroxid is added an orange-yellow color will slowly appear. The hydrogen peroxid, if not fully destroyed, will give rise to a purple color of gradual formation. The cherry or raspberry red produced as a result of methyl alcohol appears quickly after the addition of the reagent, and fades quickly unless quite intense. The intensity of the red color is in proportion to the quantity of methyl alcohol present. If the wood alcohol be as much as 1 part to 20 of ethyl alcohol, its presence will be revealed by this test.

For comparison, test the ten drops reserved in the casserole for formaldehyde after the addition of 10 cc of milk by Leach's modification of Hehner's test as directed on page 108, using care not to boil the mixture.^a

(b) REMOVAL OF FORMALDEHYDE BY METHOD OF S. P. MULLIKEN.^b

Oxidize 5 cc of the diluted alcohol as directed above. Add 1 cc of strong ammonia to the oxidized liquid in a casserole and expel the acetaldehyde by boiling gently over a direct flame until the vapor ceases to smell of ammonia. Add 2 to 3 drops of strong hydrochloric acid to set free the formaldehyde which has been retained as hexamethyltetramin, and bring the liquid momentarily to a boil; cool promptly by immersion of the casserole in water and test for formaldehyde by the modified resorcin test,^c as follows:

Add to the liquid remaining 1 drop of a solution containing 1 part of resorcin in 200 parts of water, and pour the mixture cautiously into a test tube containing 3 cc of concentrated sulphuric acid, holding the tube in an inclined position in such a manner that the two liquids shall not mix. Allow it to stand 3 minutes, then sway the tube slowly from side to side in such a manner as to produce a gentle rotary motion of the two layers. Persist in this operation, if necessary, for a minute or more, using a piece of white paper for a background, and producing only a very gradual and partial mixing of the acid and water. Nearly half of the acid should remain as a distinct unmixed layer at the end. When methyl alcohol is present, the shaking causes the separation of more or less voluminous flocks of a very characteristic rose-red color. The appearance of colored zones or flocks of other hues, even when tinged with red, or of a rose-red solution without flocks, should never be considered proof of the presence of methyl alcohol. However, if the flocks are reddish-brown, or if the upper layer has a pronounced red, it is often well to repeat the test. By this method for the removal of acetaldehyde 10 per cent of methyl alcohol may be readily detected, and an experienced operator may detect with certainty a less amount.^d

7.—DETERMINATION OF LEMON OIL.

(a) BY POLARIZATION.

Polarize the extract without dilution in a 200-mm tube at a temperature of 20° C., using the sugar scale. Divide the reading by 3.2 and, in the absence of other optically active substances, the result will be the percentage of lemon oil by volume.^e

A small amount of cane sugar is occasionally present, being used to facilitate solu-

^a Mulliken and Scudder advise against the use of the casein test owing to its extreme delicacy and to the fact that minute amounts of formaldehyde may be produced by the oxidation of grain alcohol. *Amer. Chem. Jour.*, 1900, **24**, 444.

^b Personal communication from A. G. Woodman.

^c *Amer. Chem. Jour.*, 1899, **21**, 266.

^d In the examination of other alcoholic liquids the substances interfering with the resorcin test, together with methods for their removal, may be found by consulting the original article. *Amer. Chem. Jour.*, 1899, **21**, 266.

^e J. C. Mims finds pure oil of lemon to polarize as high as 35° in solutions of 10 per cent by volume.

tion of the oil. In such case wash the "solid residue" from 10 cc of sample with three portions of 5 cc each of ether to remove waxy and fatty matters; dry and weigh residue of cane sugar, deducting 0.38 from the reading for each 0.1 per cent of sugar so found.

(b) BY PRECIPITATION.

Pipette 20 cc of the extract into a Babcock milk flask; add 1 cc dilute hydrochloric acid (1:1); add 25 to 28 cc of water previously warmed to 60° C.; mix and stand in water at 60° for five minutes; whirl in centrifuge for five minutes; fill with warm water to bring the oil into the graduated neck of the flask; repeat whirling for two minutes; stand in water at 60° for a few minutes and read the per cent of oil by volume. Where the oil of lemon is present in amounts over 2 per cent add to the percentage of oil found 0.4 per cent to correct for the oil retained in solution. Where less than 2 per cent and more than 1 per cent is present, add 0.3 per cent for correction.

Save the precipitated oil for the determination of refraction.

When the extract is made in accordance with the United States Pharmacopoeia the results by the two methods just given should agree within 0.2 per cent.

To obtain per cent by weight from per cent by volume, as found by either of the above methods, multiply the volume percentage by .980 and divide the result by the specific gravity of the original extract.

8.—DETERMINATION OF REFRACTION OF PRECIPITATED OIL.

(a) Determine the refractive index of the precipitated oil as directed under Edible Oils and Fats on page 22. Compare with similar precipitated oil from standard extract. Or,

(b) Place a few drops of the oil obtained above in a Zeiss butyro-refractometer at a temperature of 30°. Normal oil when treated under these conditions will have a refraction of 67° to 72° and a dispersion of 2°.

Limonene and most commercial adulterants give a higher reading, with the exception of citronella aldehyde and oil of turpentine.^a

9.—DETECTION OF COLORING MATTERS.

Follow directions given under coloring materials (p. 111). For Arata's test (see page 112) the "total residue" may be used after the solution in water. Upon the addition of hydrochloric acid in the determination of oil by precipitation, valuable indications as to the color are frequently given. Tartrazin, naphthol yellows, and curcuma retain their color, a pink or red coloration indicates a tropaeolin, while Martius' yellow and salts of di-nitrocresol are precipitated with decolorization of the extract.

XIII.—FRUITS AND FRUIT PRODUCTS.

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1. GENERAL DISCUSSION.

In the examination of fruits and fruit products, much depends upon the object in view; and the preparation of samples, and the determinations made will depend largely upon the judgment of the analyst. For example, in the determination of heavy metals in canned fruits, one would not be justified in using the liquor, but should work on the pulped contents of the can, while for the detection of glucose,

^aJour. Amer. Chem. Soc., 1899, 21, 1132.

preservatives, or coloring matter, examination of the liquor would be sufficient. The relative weights of liquor and fruit may be of value in detecting the presence of an excessive amount of water. A. S. Mitchell^a has noted the presence of free sulphuric acid in jellies. The methods for determination of mineral acids in vinegar can be readily applied to fruit products. The presence of phosphoric acid would be shown in the examination of the ash.

The determination of solids by drying at 100° C. gives lower results than drying in vacuo at lower temperature, or calculation from the specific gravity of the solution, the reason undoubtedly being that levulose is dehydrated at 100° C.^b But as few laboratories are equipped to use the drying in vacuo method, and as it is not possible to determine specific gravity in all cases, it is necessary to adopt some method which will give uniform results. In such a method the empirical rules have to be followed closely in order to obtain comparative results.

2. PREPARATION OF SAMPLE.

(a) JUICES, JELLIES, AND SIRUPS.

Prepare the fresh juices by pressing in a jelly bag the well pulped fruit and filtering through muslin. In the case of fresh fruit juices and fresh fruits the determination of total and volatile acids and sugars, and the polarization should be made at once, as fermentation takes place in a very short time. Portions for polarization and reducing sugar may be weighed out and an excess of lead sub-acetate added. They can then be kept for several days, if desired, without fermentation. All samples must be transferred without delay to glass-stoppered bottles and kept in a cool place.

In the case of jellies, thoroughly mix to ensure uniformity in sampling. Weigh 60 grams into a 300-cc flask, dissolve in water by means of frequent shaking, make up to the mark with water, and use aliquot portions for the various determinations. With jellies that contain starch or other insoluble material, thoroughly mix before taking aliquot portions for the various determinations.

Dealcoholize sirups by evaporation to one-third their volume and dilute with water till they contain from 15 to 20 per cent of solids.

(b) FRESH FRUITS.

Pulp the whole, well-cleaned fruit in a large mortar or by means of a food chopper and mix thoroughly. In case of stone fruits remove the pits and determine their proportion in a weighed sample.

(c) JAMS, MARMALADES, PRESERVES, AND CANNED FRUITS.

Thoroughly pulp the entire contents of the jar or can, as directed under fresh fruits; with stone fruits remove the pits, and if desired determine their proportion in a weighed sample. In the examination of canned fruits it is often sufficient to merely examine the sirups in which the fruits are preserved. In such cases the liquor may be separated and treated as is prescribed for juices.

3.—DETERMINATION OF TOTAL SOLIDS.

(a) IN JUICES, JELLIES, AND SIRUPS.

(1) *By direct determination.*—Measure 25 cc° of a 20 per cent solution [see 2 (a)] of jelly, or weigh 25 grams of juice, into a large flat-bottomed dish which contains

^a Communicated by letter.

^b Carr and Sanborn, U. S. Dept. of Agr., Division of Chemistry, Bul. 47, p. 134.

^c If a pipette be used it must be graduated so as to deliver a definite volume of a 20 per cent sugar solution after draining a definite time.

about 4 or 5 grams of freshly ignited asbestos to absorb it; dry for from 20 to 24 hours in a water-jacketed oven.^a If care is taken in measuring, this method will be found to be as accurate as weighing. In case of jellies that contain starch or insoluble matter, solids may be determined as directed below under (b).

(2) *By calculation from specific gravity.*—Determine the specific gravity of the solution of jelly or diluted sirup, or of the juice, by means of a Westphal balance, pycnometer, or specific-gravity spindle, and calculate the solids from Table IV.^b

(b) IN FRESH FRUITS, JAMS, MARMALADES, PRESERVES, AND CANNED GOODS.

Weigh about 20 grams of pulped fresh fruit, or such an amount of fruit products as will give not more than 3 to 4 grams of dried material, into a large flat-bottomed dish containing ignited asbestos; add a few cubic centimeters of water, mix thoroughly, and dry as in [(a) 1].

4.—DETERMINATION OF INSOLUBLE SOLIDS.

(a) KREMLA'S METHOD MODIFIED.

Weigh 50 grams of the sample; transfer by the aid of warm water to a mortar and thoroughly macerate,^c then transfer to a muslin filter and wash thoroughly with warm water, care being taken at each addition of water to thoroughly stir the pulp. Collect the filtrate in a 500-cc flask, cool and make up to volume. Usually this amount is sufficient to remove all soluble material. In extreme cases increase the washings to 1000 cc; transfer the insoluble residue to an evaporating dish, dry, and weigh.

(b) GERMAN OFFICIAL METHOD.

Transfer a weighed portion of the fruit product to a graduated flask, add water, shake thoroughly and make up to volume. Allow this to settle and either filter or decant off the supernatant liquid. Take an aliquot for the determination of soluble solids. Total solids less soluble solids equals insoluble solids. The fruit must be thoroughly macerated and the use of a mechanical shaker would be advisable.

5.—DETERMINATION OF ALCOHOL.

Determine alcohol in 50 grams of the original material according to the method prescribed on page 82.

6.—DETERMINATION OF ASH.

Evaporate to dryness 50 cc of the solution of jelly or diluted sirup [see 2 (a)], 25 grams of juice or fresh fruit, or 10 grams of jam, marmalade, preserves, or canned fruit in a large platinum dish; then thoroughly char at as low a heat as possible, extract with water, filter, and wash. Return the filter paper and insoluble material to the dish and thoroughly ignite; add the soluble portion and evaporate the whole to dryness after adding a few cubic centimeters of a solution of ammonium carbonate; then heat for a moment to very low redness; cool in a desiccator and weigh. The weighing must be made as quickly as possible, as the ash absorbs moisture very rapidly.

^a A. McGill, Laboratory of Internal Revenue, Ottawa, Canada, has devised a forced draft water oven for drying at temperatures between 60° and 90° C. The oven is heated by means of ordinary gas burners, and the temperature is controlled by introducing at the bottom of the oven a blast of air from a blower run by a small water motor. Before discharging into the oven the air tube enters the water chamber and is coiled a number of times in order to sufficiently warm the water before it enters the oven. The exit end of the air tube is covered with a concave-convex disc in order to distribute the blast and to prevent harmful currents. By regulating the burners and the flow of air a fairly constant temperature can be obtained. The bottom of the oven is curved instead of flat, to prevent bumping when the water is boiling; a perforated plate serves as a false bottom.

^b Ztschr. Nahr. Hyg. Waar. 1892, 6, 483.

^c McGill, by letter, recommends the use of a mechanical shaker to obtain complete solution of the soluble material.

7.—EXAMINATION OF ASH.

(a) ALKALINITY OF THE ASH.

Into the platinum dish containing the ash run an excess of fifth-normal nitric acid and add a few drops of methyl orange. Carefully rub up the ash with a rubber tipped stirring rod and titrate the excess of acid with decinormal potassium hydroxid. Calculate the alkalinity to per cent of potassium carbonate in the original substance. One cubic centimeter of decinormal acid equals 0.00691 gram of potassium carbonate.

(b) SULPHATES AND CHLORIDS.

Wash the ash into a 50-cc flask and make up to the mark with water. In 25 cc of this solution determine the sulphates by precipitation with barium chlorid. The weight of barium sulphate times 0.7478 gives the weight of sulphates calculated as potassium sulphate.

In the other portion of the solution determine the chlorids by the Volhard^a method for chlorin. The nitric acid added before making the titration will, if it contain enough nitrous oxid, completely destroy the red color of the methyl orange and leave a clear solution for the titration. Calculate the chlorid as per cent of sodium chlorid. Pure fruit jellies and jams give practically no chlorids or sulphates in this amount of ash, but glucose goods give appreciable amounts. If it is desired to make a complete ash analysis of juices or fresh fruits much larger amounts will have to be ashed.

8.—DETERMINATION OF TOTAL ACIDITY.

Take 25 cc of the solution of jelly or diluted sirup [see 2 (a)], 10 grams of juice or fresh fruit, or 50 cc of the washings from the determination of insoluble solids, and dilute with recently boiled distilled water to about 250 cc, or less if the jelly be not highly colored; add phenolphthalein and titrate the acid with decinormal potassium hydroxid. In case of highly colored products litmus paper may be used instead of phenolphthalein. Calculate the results as sulphuric acid.^b

It is very desirable that acidity be so expressed as to allow of comparison. This end is not attained by expressing the acidity in terms of the dominant acid of the various fruits; hence H_2SO_4 has been suggested, and already a number of laboratories have used this as a basis.

9.—DETERMINATION OF VOLATILE ACIDS.

The determination of volatile acids in fruit products may be desirable in cases where fermentation or the use of decayed fruit is suspected. Dissolve 25 grams of substance, dilute to 50 cc, and distill in a current of steam until about 200-cc have passed over. Titrate the distillate with decinormal potassium hydroxid and express the results as acetic acid. Each cubic centimeter of decinormal alkali is equivalent to 0.006 gram of acetic acid.

10.—DETECTION OF FREE MINERAL ACIDS.

A. S. Mitchell and A. E. Leach have both called attention to the presence of free sulphuric and phosphoric acid in jellies. For method of detection see Hehner's method, page 64.

11.—DETERMINATION OF NITROGEN.

Use 5 grams of jelly or other fruit product or 10 grams of juice or fresh fruit for the determination of nitrogen according to either the Gunning or the Kjeldahl method. Express results as protein (nitrogen multiplied by 6.25).

^a Ann. d. Chem. 1877, 190, 1. Sutton, Volumetric Analyses, eighth edition, p. 155.

^b See Composition of Jellies and Jams. Tolman, Munson, and Bigelow. Jour. Am. Chem. Soc. 1901, 23, 348.

12.—POLARIZATION.

Dissolve half the normal weight of jelly or other fruit product, or the normal weight of juices or fresh fruits, in a sufficient quantity of water in a 100-cc sugar flask, add an excess of lead subacetate (from 5 to 10 cc, see footnote, p. 84), filter, and polarize in a 200-mm tube, observing the temperature of the solution. Invert 50 cc of this solution using 5 cc of hydrochloric acid and heating to 68° C. in 15 minutes. Polarize in a 220-mm tube at the same temperature as was employed in making the direct reading.

On account of the large amounts of invert sugar usually found in these products it is necessary that the direct and invert readings should be made at the same temperature.

13.—DETERMINATION OF CANE SUGAR.

(a) BY POLARIZATION.

Calculate cane sugar from the direct and the invert readings according to Clerget's formula:

$$S = \frac{100 (a - b)}{144 - \frac{t}{2}}$$

(b) BY INVERSION.

Where only a small amount of cane sugar is present it is best determined by calculation from the increase in reducing sugars after inversion. For this purpose treat 5 grams of jelly, sirup, or other fruit product, or 25 grams of juice or fresh fruit with lead subacetate in excess, and after making up to 100 cc and filtering invert 50 cc in a 100-cc flask with 5 cc of hydrochloric acid. After inversion neutralize the acid with sodium hydroxid, precipitate excess of lead with sodium sulphate and increase in volume to 100 cc. Filter and dilute so that the solution does not contain more than 1 per cent of reducing sugar. The per cent increase in reducing sugar after inversion multiplied by 0.95 equals per cent of cane sugar.

14.—DETERMINATION OF REDUCING SUGARS.

Treat 5 grams of jelly (25 cc of a 20 per cent solution [2 (a)] may be employed), sirup, or other fruit product, or 25 grams of juice or fresh fruit with lead subacetate in excess (2 to 5 cc); make up to 100 cc and filter. Transfer from 25 to 50 cc—depending upon the per cent of reducing sugar present—to a 100-cc flask and add a saturated solution of sodium sulphate in sufficient amount to precipitate the excess of lead; complete the volume to 100 cc and use the filtered solution for the determination of reducing sugars. The approximate amount of reducing sugar present may be readily ascertained from the polarizations and from the percentage of solids. Use Allihn's method for the determination (p. 49).^a

15.—DETERMINATION OF DEXTRIN.

Dissolve 10 grams of the sample^b in a 100-cc flask; add 20 mg of potassium fluorid and then about one-quarter of a cake of compressed yeast.^c Allow the fermentation to proceed below 25° C. for 2 or 3 hours to prevent excessive foaming, and then place in an incubator at a temperature of from 27° to 30° C. for 5 days. At the end of that time, clarify with lead subacetate and alumina cream; make up to 100 cc and polarize in a 200-mm tube. A pure fruit jelly will show a rotation of not more than a few

^a U. S. Department of Agriculture, Division of Chemistry, Bulletin 46 revised, page 35.

^b In the case of jellies, 50 cc of a 20 per cent solution, prepared as directed under 1 (a), may be used.

^c Bigelow and McElroy. Jour. Am. Chem Soc. 1893, 15, 668.

tenths of a degree either to the right or to the left.^a If a Schmidt and Haensch polariscope be used and a 10 per cent solution be polarized in a 200-mm tube, the number of degrees read on the sugar scale of the instrument multiplied by 0.8755 will give the percentage of dextrin, or the following formula^b may be used:

$$\text{Percentage of dextrin} = \frac{C \times 1,000 \times V}{198 \times L \times W}$$

in which

C=degrees of circular rotation,

V=volume in cubic centimeters of solution polarized,

L=length of tube in centimeters,

W=weight of sample in solution in grams.

16.—DETERMINATION OF ALCOHOL PRECIPITATE.

Take 100 cc of a 20 per cent solution of jelly [see 2 (a)], diluted sirup, or of the washings from the determination of insoluble solids, and evaporate to 20 cc; then add slowly and with constant stirring 200 cc of 95 to 96 per cent alcohol and allow the mixture to stand overnight. Filter and wash with 80 per cent alcohol by volume. Wash this precipitate off the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100° C. for several hours and weigh; then burn off the organic matter and weigh the residue as ash. The loss in weight upon ignition is called alcohol precipitate.

The ash should be largely lime and not more than 5 per cent of the total weight of the alcohol precipitate. If it is larger than this some of the salts of the organic acids have been brought down. Titrate the water-soluble portion of this ash with decinormal acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

The general appearance of the alcohol precipitate is one of the best indications as to the presence of glucose and dextrin. Upon the addition of alcohol to a pure fruit product a flocculent precipitate is formed with no turbidity, while in the presence of glucose a white turbidity appears at once upon adding the alcohol, and a thick, gummy precipitate forms.

17.—DETERMINATION OF TARTARIC, CITRIC, AND MALIC ACIDS.^c

Use the filtrate from the alcohol precipitate in the determination of the organic acids. After evaporating off the alcohol and taking up the acids with water add lead subacetate until the solution is alkaline, then filter and wash the precipitate until only a slight amount of lead remains in the washings. Wash the precipitate off the filter paper into a beaker with hot water, precipitate the lead by hydrogen sulphid and filter off the lead sulphid while hot, washing with hot water. Evaporate the filtrate which contains the free organic acids to about 50 cc, neutralize exactly with potassium hydroxid, add an excess of strong solution of neutral calcium acetate with constant stirring, and allow to stand from 6 to 12 hours. Throw the precipitate of calcium tartrate on a filter paper and wash until filtrate and washings make exactly 100 cc; ignite the filter paper and precipitate, and determine the lime and tartaric acid by titration. A correction of 0.0286 grams of tartaric acid, which is held in solution in the 100 cc of washings as calcium tartrate, must be added. Now evaporate the filtrate down to about 20 cc, and if a precipitate of calcium citrate is formed filter it off hot, wash with hot water, ignite, and titrate the lime. From

^a U. S. Dept. of Agr., Div. of Chem., Bul. 66.

^b Wiley, Chem. News, 1882, 46, 175.

^c A modification of Schmidt & Hiepe's method. U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 67; Ztschr. anal. Chem., 1882, 21, 534-541.

this calculate the citric acid. Again evaporate the filtrate to about 20 cc and add 3 volumes of 96 per cent alcohol by volume, which will throw down the calcium salt of tartaric acid held in solution, the rest of the citrate, and the malate and succinate. Filter this off, ignite, titrate, and calculate as malic acid, after subtracting the tartaric acid present, as the amount of citric and succinic acid present is very small.

18.—DETERMINATION OF TARTARIC ACID.^a

To 100 cc of the fruit juice add 2 cc of glacial acetic acid, 2 or 3 drops of a 20 per cent potassium acetate solution and 15 grams of pure finely powdered potassium chlorid, dissolve this by shaking, and then add 20 cc of 96 per cent alcohol. Then stir vigorously for one minute, rubbing the walls of the beaker with the glass stirring rod to start the crystallization of the potassium bitartrate. Allow to stand 15 hours at room temperature. Filter and wash the precipitate onto a Gooch crucible with a thin asbestos felt, using the vacuum pump. Wash with a mixture of 15 grams of potassium chlorid, 20 cc alcohol, and 100 cc water. The beaker is rinsed three times with a few cubic centimeters of this solution. The precipitate is also washed with a few cubic centimeters, but so that not more than 20 cc in all of the wash solution is used. The precipitate and asbestos filter are washed back into the beaker and heated to boiling. While still hot the solution is titrated with decinormal alkali, using phenolphthalein as indicator. To the amount of alkali used must be added 15 cc for the potassium bitartrate remaining dissolved in the solution. One cubic centimeter of decinormal alkali is equivalent to .0150 grams potassium bitartrate.

19.—DETERMINATION OF CITRIC ACID.^b

Fifty cubic centimeters of the fruit solution is evaporated on the water bath to a sirupy condition. To the residue add, very slowly at first, stirring constantly, 95 per cent alcohol until no further precipitate is formed; 70 to 80 cc are generally enough. Filter and wash the residue with 95 per cent alcohol. Evaporate the filtrate to eliminate the alcohol, take up the residue with a little water and transfer to a graduated cylinder, making up to 10 cc. To 5 cc of this solution add half a cubic centimeter of glacial acetic acid, and to this add, drop by drop, a saturated solution of lead acetate. The presence of citric acid is shown by the appearance of a precipitate which possesses the property of disappearing on being heated and reappearing on cooling. In order to separate the citric acid from other acids, heat to boiling, filter, and wash with boiling water; then allow to cool and the precipitate of lead citrate will re-form. This lead precipitate may be filtered off, washed into weak alcohol, dried, weighed, and the citric acid calculated. It is necessary that there shall be no tartaric acid present. If the tartaric acid has been estimated, any error on this account may be avoided by adding enough decinormal potash to neutralize the tartaric acid before the alcohol is added.

20.—DETECTION OF PRESERVATIVES.

Dissolve about 25 grams of the sample in water, acidify, and extract with ether. Remove the ether layer and allow it to evaporate spontaneously. Take up the residue, which may contain salicylic and benzoic acids and saccharin, with water. For detecting preservatives so separated, and to test further for preservatives, use methods described by the referee on that subject (p. 107).

21.—DETECTION OF COLORING MATTER.

Follow directions given on pages 111 and following.

22.—DETECTION OF ARTIFICIAL SWEETENING MATERIALS.

Follow directions given on page 89.

^a Halenke & Möslinger, *Ztschr. anal. Chem.*, 1895, **34**, 283.

^b Möslinger, *Ztschr. Unter. Nahr. u. Genuss.*, 1899, **2**: 93.

23.—DETECTION OF STARCH.

First destroy the color of the jelly by treatment with sulphuric acid and potassium permanganate and then test with iodine. Bring the solution of jelly nearly to the point of boiling, add several cubic centimeters of dilute sulphuric acid and then potassium permanganate until all color is destroyed. By this treatment the starch remains unaffected. The test for starch is not necessarily an indication of its addition as an adulterant. It is almost always present in the apple, and occasionally in other fruits, and unless it is present in the jelly or other fruit product in considerable amounts it may be due to that source.

24.—DETECTION OF GELATIN.

The presence of gelatin in jellies and jams is shown by a higher content of nitrogen. Precipitate a concentrated solution of jelly or jam with 10 volumes of absolute alcohol and determine nitrogen in dried precipitate by the Gunning method.^a

25.—DETECTION OF AGAR AGAR.^b

Cook the jelly with 5 per cent sulphuric acid, add a crystal of potassium permanganate and allow to settle. If agar is present the sediment will be rich in diatoms, which can be detected by use of microscope.

26.—THE DETERMINATION OF HEAVY METALS.

Treat 100 grams of the preserve directly in a large porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. If much water is present evaporate the material to a sirupy consistency before treating with the acid. From 15 to 25 cc of strong acid has been found sufficient to thoroughly carbonize the amount specified. Then ash the material, transfer the ash to a beaker of about 400-cc capacity, slightly acidify it with hydrochloric acid, and boil for a few moments. Methods for separation and determination of metals are given on page 52.

XIV.—FERMENTED AND DISTILLED LIQUORS.

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(A) WINE.

The determinations of most value in judging the purity of wine are alcohol, glycerol, extract, ash, total and volatile acids, and reducing sugar. The actual percentage of these substances present is of interest, but much more important are certain relations between them, such as ash to extract, extract to alcohol, alcohol to glycerol, alcohol to acids, and volatile acids to total acids. Examination for preservatives and foreign coloring matter must also be made. Search is sometimes made for lead, which may result from cleansing bottles with the aid of shot; for copper and arsenic, which sometimes result from the use of insecticides and fungicides on the grapes; and for barium and strontium, which are sometimes used in southern Europe to remove the excess of sulphate introduced by plastering. A qualitative test is often made for nitrates to detect the addition of (impure) water, and for dextrin to determine whether glucose has been used in the preparation of the wine.

Information regarding the manner of preparing the wine is often afforded by the determination of tannin, potassium sulphate (for plastered wines), and of phosphoric acid.

^a A. Boemer, Chem. Ztg., 1895, 19, 552.

^b C. Marpmann. Ztschr. f. angew. Mikrosk. 1896, 2, 260.

For the various determinations, a measured volume can be taken more conveniently than a weighed quantity. The results can be calculated to per cent by weight by dividing the results expressed as grams per 100 cc by the specific gravity.

1.—DETERMINATION OF SPECIFIC GRAVITY.

Determine specific gravity at the temperature of 15.6° C. by means of the pycnometer, small accurately graduated hydrometer, Westphal balance, or a Westphal plummet on the analytical balance. The pycnometer, when used, should be heated quickly to room temperature after filling and before weighing, to prevent the error due to the collection of moisture on the outside. A small hole filed in the cap will permit the necessary expansion in the volume of liquid.

2.—DETERMINATION OF ALCOHOL.

Measure 100 cc of the liquid into an Erlenmeyer flask of from 250 to 300-cc capacity; add 50 cc of water; attach the flask to a vertical condenser by means of a bent tube and distill 100 cc. Foaming, which sometimes occurs, especially with new wines, may be prevented by the addition of a small amount of tannin. If it be desired to determine alcohol in wines which have undergone acetic fermentation and contain a large amount of acetic acid, 0.1 or 0.2 gram of precipitated calcium carbonate should be added. This is unnecessary, however, in wines of normal taste and odor. Where only occasional determinations of alcohol are made it is found convenient to use an alembic Saleron. This apparatus is made of copper, and it can be readily taken apart and placed in a small box. No rubber connections are necessary, and the setting up requires but a few minutes. Determine the specific gravity of the distillate as directed under Specific Gravity, and obtain the corresponding percentage of alcohol, by volume and grams per 100 cc, from Table II.

3.—DETERMINATION OF GLYCEROL.

Evaporate 100 cc of wine^a in a porcelain dish on the water bath to a volume of about 10 cc and treat the residue with about 5 grams of fine sand and with from 1.5 to 2 cc of milk of lime (containing about 40 per cent of calcium hydroxid or 30 per cent of calcium oxid) for each gram of extract present, and evaporate almost to dryness. Treat the moist residue with 5 cc of 96 per cent alcohol (sp. gr. 0.81), remove the substance adhering to the sides of the dish with a spatula, and rub the whole mass to a paste, with the addition of a little more alcohol. Heat the mixture on the water bath, with constant stirring, to incipient boiling, and decant the liquid into a flask graduated at 100 and 110 cc. Wash the residue repeatedly by decantation with 10 cc portions of hot 96 per cent alcohol. Cool the contents of the flask to 15°, dilute to the 110-cc mark with 96 per cent alcohol, and filter through a folded filter. Evaporate 100 cc of the filtrate to a sirupy consistency in a porcelain dish, on a hot, but not boiling, water bath, transfer the residue to a small glass-stoppered graduated cylinder with 20 cc of absolute alcohol, and add three portions of 10 cc each of absolute ether, mixing after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with a mixture of one part absolute alcohol to one and one-half parts of absolute ether, pouring the wash liquor also through the filter. Evaporate the filtrate to a sirupy consistency, dry for one hour at the temperature of boiling water, weigh, ignite, and weigh again. The loss on ignition increased by one-tenth gives the glycerol expressed in grams per 100 cc.

^a With wines whose extract exceeds 5 grams per 100 cc, heat to boiling in a flask the portion to be used in the determination of glycerol, and treat with successive small portions of milk of lime until it becomes, first, darker, and then light in color. When cool, add 200 cc of 96 per cent alcohol (sp. gr. 0.8118), allow the precipitate to subside, filter, and wash with 96 per cent alcohol (sp. gr. 0.8118). Evaporate the filtrate to about 10 cc, add about 5 grams of sand and from 1.5 to 2 cc of milk of lime and proceed as directed above.

4.—DETERMINATION OF EXTRACT.

(a) FROM SPECIFIC GRAVITY OF DEALCOHOLIZED WINE.

Preliminary to its exact determination, the extract should be calculated by the formula:

$$sp = 1 + x - x'$$

in which sp is the specific gravity of the dealcoholized wine, x the specific gravity of the wine, x' the specific gravity of the alcoholic distillate obtained in the estimation of alcohol.

Illustration.—A sample of Catawba is examined with the result:

Specific gravity of wine (x)	1.0402
Specific gravity of alcoholic distillate (x')	.9857
Difference ($x - x'$)	.0545
Specific gravity dealcoholized wine ($1 + x - x'$)	1.0545
Extract (from Table V)	14.48 grams per 100 cc.

The extract content equivalent to sp is obtained from table.

(b) BY EVAPORATION.

(1) *In dry wines.*

(Having an extract content of less than 3 grams per 100 cc).

Evaporate 50 cc of the sample on the water bath to a sirupy consistence in a flat-bottom platinum dish about 85 mm in diameter and capable of holding about 75 cc. Heat the residue for two and a half hours in a drying oven at the temperature of boiling water and weigh. This weight multiplied by 2 gives grams of total residue in 100 cc. The sugar-free extract is found by deducting the weight of sugar in excess of 0.1 gram per 100 cc from the total residue. In the case of plastered wines, the potassium sulphate in excess of 0.1 gram is also deducted.

(2) *In sweet wines.*

When the extract content is between 3 and 6 grams per 100 cc treat 25 cc of the sample as described under dry wines. When the extract exceeds 6 grams per 100 cc, however, the result obtained under (a) is accepted, and it is not attempted to determine it gravimetrically. This is because of the serious error connected with drying levulose at high temperature. The table referred to here was obtained by drying at 75° C. in vacuo.

5.—DETERMINATION OF ASH.

Ignite at low redness, until thoroughly charred, the residue from the determination of extract,^a exhaust with water, filter, and wash. Return the filter paper and insoluble material to the dish and burn to a white ash, add the soluble portion and evaporate the whole to dryness, heat to a low redness, cool in a desiccator, and weigh. With dry wines complete combustion can often be obtained without leaching.

6.—DETERMINATION OF TOTAL ACIDS.

Expel any carbon dioxid that is present by continued shaking. Transfer 25 cc of the sample to a beaker, heat to incipient boiling, and, in the case of white wines, add about 10 drops of a neutral litmus solution and titrate while still hot with decinormal sodium hydroxid solution. With red wines, add decinormal sodium hydroxid solution until the red color changes to violet, and continue adding a few drops at a time until a drop of the mixture placed on delicate neutral litmus paper ceases to show an acid reaction.^b The result is expressed in terms of tartaric acid.

^a Employ the residue obtained by evaporating 25 cc of the wine when the extract has been calculated from specific gravity.

^b See Appendix, p. 155.

One cubic centimeter of decinormal sodium hydroxid solution is equivalent to 0.0075 gram tartaric acid.

7.—DETERMINATION OF VOLATILE ACIDS.

Distill, in a current of steam, 50 cc of wine, to which a little tannin has been added to prevent foaming. Heat the flask containing the sample until the liquid boils, lower the flame under it and pass the steam through until 200 cc have been collected in the receiver; titrate the distillate with decinormal sodium hydroxid solution, using phenolphthalein as indicator, and express the result as acetic acid.

One cubic centimeter of decinormal sodium hydroxid solution is equivalent to 0.006 gram acetic acid.

8.—DETERMINATION OF FIXED ACIDS.

The amount of fixed acids is ascertained by subtracting 1.25 times the volatile acids from the total acids expressed as tartaric.

9.—DETERMINATION OF UNDETERMINED EXTRACT.

The amount of undetermined extract is ascertained by subtracting the sum of the glycerol, ash, protein, and fixed acids from the weight of the sugar-free extract.

10.—DETERMINATION OF SUGAR.^a

(a) PREPARATION OF SOLUTION.

Place 200 cc of wine in a porcelain dish, exactly neutralize with an approximately normal solution of sodium hydroxid, using litmus paper as indicator, and evaporate to about one-fourth the original volume. Transfer to a 200-cc flask, add sufficient basic lead acetate^b to clarify, dilute to the mark with water, shake, and filter through a ribbed filter. Transfer 100 cc of the filtrate to a flask graduated at 100 and 110 cc, fill to the upper mark with a saturated solution of sodium sulphate, shake and filter.

(b) POLARIZATION.

(1) *Direct.*

Polarize part of the filtrate in a 200-mm tube, in a Schmidt and Haensch polariscope, and increase the reading by one-tenth for the polariscope reading. In case the reading is taken on some other instrument than the Schmidt and Haensch it may be calculated by the following data:

1° Ventzke	=0.3468° angular rotation D.
1° angular rotation D	=2.8835° Ventzke.
1° Ventzke	=2.6048° Wild (sugar scale).
1° Wild (sugar scale)	=0.3840° Ventzke.
1° Wild (sugar scale)	=0.1331° angular rotation D.
1° angular rotation D	=0.7511° Wild (sugar scale).
1° Laurent (sugar scale)	=0.2167° angular rotation D.
1° angular rotation D	=4.6154° Laurent (sugar scale).

(2) *Invert.*

In order to determine the presence or absence of sucrose it is necessary to subject the sugars to inversion. The filtrate from the lead sulphate obtained in (a) may be

^a See Appendix, p. 156.

^b Prepared by boiling for half an hour 430 grams of normal lead acetate, 130 grams of litharge, and 1000 cc of water. The mixture is allowed to cool and settle. when the supernatant liquid is diluted to 1.25 specific gravity with recently boiled water

conveniently employed for this purpose. Fill a flask graduated at 50 and 55 cc to the 50-cc mark with the filtrate, add 5 cc of concentrated hydrochloric acid, invert; polarize in a 220-mm tube, and increase the reading one-tenth to allow for dilution.

(3) *After fermentation.*

In the case of wines polarizing between $+2.3^{\circ}$ and $+0.9^{\circ}$ the use of glucose in their preparation can be proved or disproved after fermentation by the presence or absence of certain unfermentable constituents.

Dealcoholize 200 cc of wine by evaporating to about one-fourth its volume, and add enough water to the residue to make its sugar content less than 15 per cent. For the purpose of this operation the sugar content of the wine may be assumed to be 2 per cent less than the extract. Add 2 or 3 grams of compressed yeast, let stand at about 25° C. for four or five days, when fermentation will be complete.

Evaporate the fermented liquid in a porcelain dish to a thin sirup after the addition of a little sand and a few drops of a 20 per cent solution of potassium acetate. To the residue add 200 cc of 90 per cent alcohol with constant stirring. Separate the alcoholic solution by filtration and evaporate until about 5 cc remain. Mix the residue with washed boneblack, filter into a graduated cylinder, and wash until the filtrate (cooled to 15° C.) amounts to 30 cc. When the filtrate shows a dextrorotation of more than 1.5° it indicates the presence of the unfermentable constituents of commercial glucose. Results by this method are not reliable with wines that are heavily preserved.

(c) REDUCING SUGARS.

Dilute a portion of the solution prepared as directed under (a) until it does not contain more than 1 per cent of sugar. In making this dilution the sugar-free extract of a wine may be taken as 2 per cent. The number of volumes of water to be added to the filtrate is thus determined by deducting 2 from the total extract. If the wine is not to be polarized, or for any reason a separate portion is to be prepared for the reduction, the dilution may be conveniently made prior to the clarification. Use Allihn's method, expressing the results as dextrose (see p. 49).

(d) CANE SUGAR.

(1) *By reduction.*

Invert a portion of the filtrate obtained in (a) as directed under (b) (2); determine reducing sugars according to (c); deduct from the figure thus obtained the reducing sugars originally present, and multiply the result by 0.95 for conversion into cane sugar.

(2) *By polarization.*

Calculate from the direct and invert polarizations by the Clerget formula:

$$\text{Per cent sucrose} = \frac{100 (R - R')}{144 - \frac{T}{2}}$$

(e) COMMERCIAL GLUCOSE. ^a

(1) Wine with not more than 0.1 per cent of reducing sugar, and which polarizes to the left or not more than 0.9° to the right, has not been treated with glucose.

(2) Wine with not more than 0.1 per cent of reducing sugar, and which polarizes 0.9° or more to the right, may contain dextrin and the unfermentable constituents of commercial glucose. In such a case, examine according to 10 (3) and 11.

(3) If the reducing sugar exceeds 0.1 per cent, examine according to 10 (3) for the unfermentable constituents of commercial glucose.

11.—DETERMINATION OF GUM AND DEXTRIN.

Evaporate 100 cc of wine to about 10 cc and add 10 cc of 96 per cent alcohol (sp. gr. 0.81). If gum or dextrin be present (indicated by the formation of a voluminous precipitate), continue the addition of alcohol slowly and with stirring until 100 cc have been added. Let stand over night, filter, and wash with 80 per cent alcohol by volume (sp. gr. 0.84). The precipitate may then be dried and weighed, or it may be treated according to Sachsse's method for the determination of starch.

12.—DETERMINATION OF TANNIN AND COLORING MATTER.^a

Dealcoholize 100 cc by evaporation and dilute with water to the original volume. Transfer 10 cc to a porcelain dish of about 2 liters capacity; add about a liter of water and exactly 20 cc of indigo^b solution, measuring the latter by means of a burette. Add decinormal potassium permanganate solution, which has been standardized against decinormal oxalic acid, a cubic centimeter at a time, until the blue color changes to green; then a few drops at a time until the color becomes bright yellow. Designate the number of cubic centimeters of permanganate solution employed by (a).

Treat 10 cc of the dealcoholized wine, prepared as above, with carefully purified boneblack for fifteen minutes; filter and wash the boneblack thoroughly with water. Add a liter of water and 20 cc of indigo solution and titrate with permanganate as above. Designate the number of cubic centimeters of permanganate solution employed by (b).

Then $a - b = c$ = the number of cubic centimeters of permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc of wine.

Multiply (c) (corrected to cubic centimeters of decinormal solution, if the solution employed is not exactly decinormal) by 0.04157 for tannin and coloring matter, expressed in grams per 100 cc.

One cubic centimeter of decinormal permanganate solution is equivalent to 0.004157 gram tannin.

13.—DETERMINATION OF SODIUM CHLORID.

Sodium chlorid is obtained by dissolving the ash in water, slightly acidifying with nitric acid, neutralizing with calcium carbonate, and titrating with silver nitrate, using normal potassium chromate as indicator.

14.—DETERMINATION OF POTASSIUM SULPHATE.

Precipitate sulphuric acid directly in 50 cc of wine by means of barium chlorid, and determine the resulting barium sulphate by the ordinary method. Express the result in grams of potassium sulphate per 100 cc. In all cases this determination should be made in the original wine, as results obtained with the ash are always low.

15.—DETERMINATION OF PHOSPHORIC ACID.

Determine phosphoric acid in the ash by the official volumetric method. In case the ash has been used for other determinations, and it is necessary to begin with the original wine, evaporate 100 cc of dry wines and ignite directly. With sweet wines, evaporate 100 cc to a sirupy consistency in a flask of about 250-cc capacity, add 25 cc of concentrated sulphuric acid and heat with a low flame till the evolution of gas

^a Neubauer-Löwenthal method. *Annalen der Oenologie*, 2, 1.

^b Instead of the indigo-carmin called for by the original method, sodium sulphindigotate may be employed, as suggested by Schroeder (*Ztschr. anal. Chem.*, 1886, 25, 112). To prepare the solution, dissolve 6 grams of sodium sulphindigotate in 500 cc of water with aid of heat; cool; add 50 cc of concentrated sulphuric acid; dilute to 1 liter and filter. (*U. S. Dept. of Agr., Bul. 46 revised, p. 66.*)

ceases. Add about 75 cc concentrated nitric acid, warm gently, and finally evaporate almost to dryness. Then add 10 cc of concentrated sulphuric acid and a little mercury and boil till the solution clears.^a Employ the official volumetric method for phosphoric acid, as stated above.

16.—DETERMINATION OF TARTARIC ACID AND TARTRATES.

(a) TOTAL TARTARIC ACID.^b

To 100 cc of wine add 2 cc of glacial acetic acid, 3 drops of a 20 per cent solution of potassium acetate, and 15 grams of powdered potassium chlorid, and stir to hasten solution. Add 15 cc of 95 per cent alcohol (sp. gr. 0.81) and rub the side of the beaker vigorously with a glass rod for about 1 minute to start crystallization. Let stand at least 15 hours at room temperature; decant the liquid from the separated acid potassium tartrate as rapidly as possible (using vacuum) through a Gooch crucible prepared with a very thin film of asbestos, transferring no more of the precipitate to the crucible than necessary. Wash the precipitate and filter three times with a small amount of a mixture of 15 grams potassium chlorid, 20 cc of 95 per cent alcohol (sp. gr. 0.81), and 100 cc water, using not more than 20 cc of the wash solution in all. Transfer the asbestos film and precipitate to the beaker in which the precipitation took place, wash out the Gooch crucible with hot water, add about 50 cc of hot water, heat to boiling, and titrate the hot solution with decinormal sodium hydroxid, using delicate litmus tincture or litmus paper as indicator. Increase the number of cubic centimeters of decinormal alkali employed by 1.5 on account of the solubility of the precipitate. This figure multiplied by 0.015 gives the amount of total tartaric acid in grams per 100 cc.

(b) CREAM OF TARTAR.

Ignite the residue obtained from the evaporation of 50 cc of wine as directed under the determination of ash. Exhaust the ash with hot water, add to the filtrate 25 cc of decinormal hydrochloric acid, heat to incipient boiling and titrate with decinormal alkali solution, using litmus as indicator. Deduct from 25 cc the number of cubic centimeters of decinormal alkali employed and multiply the remainder by 0.0188 for potassium bitartrate expressed in grams.

(c) FREE TARTARIC ACID.

Add 25 cc of decinormal hydrochloric acid to the ash of 50 cc of wine, heat to incipient boiling and titrate with decinormal sodium hydroxid, using litmus as indicator. Deduct the number of cubic centimeters of alkali employed from 25 and multiply the remainder by 0.0075 to obtain the amount of tartaric acid necessary to combine with all the ash (considering it to consist entirely of potash). Deduct the figure so obtained from the total tartaric acid for the free tartaric acid.

17.—DETERMINATION OF PROTEIN.

Determine nitrogen in 50 cc of wine by the Kjeldahl or the Gunning method, and multiply the result so obtained by 6.25.

18.—DETERMINATION OF HEAVY METALS.

Lead is often found in wine as a result of the use of shot in cleaning bottles, and copper and arsenic may occur in wine made from grapes sprayed with insecticides. Lead and copper may be determined in 500 or 1,000 cc by the method given under Vegetables.

^a Glaser and Mühle, Chem. Ztg., 1896, 20, 723.

^b Halenke and Möslinger, Ztschr. anal. Chem., 1895, 34, 263.

Arsenic may be detected or determined by the Marsh apparatus if combustion be effected by the method given under the determination of phosphoric acid (p. 86).

Copper may be precipitated electrolytically^a in 500 cc of the undiluted wine by using as electrodes pieces of platinum foil 3 by 15 cm.

19.—DETERMINATION OF BARIUM AND STRONTIUM.^b

Evaporate to dryness 100 cc of wine, incinerate as directed under the determination of ash (p. 83), dissolve in dilute hydrochloric acid, evaporate to dryness, and examine the residue spectroscopically. If barium or strontium be present, fuse with sodium carbonate^c to decompose silicates, dissolve in water and determine by precipitation with sulphuric acid.

20.—DETECTION OF FOREIGN COLORING MATTER.

Follow directions given under Coloring Matter (pp. 111 and following).

21.—DETECTION OF NITRATES.

(a) WHITE WINE.

Treat a few drops of the wine in a porcelain dish with 2 or 3 cc of concentrated sulphuric acid which contains about 0.1 gram of diphenylamin^d per 100 cc. The deep blue color formed in the presence of nitrates appears so quickly that it is not obscured, even in sweet wine, by the blackening produced by the action of sulphuric acid on the sugar.

(b) RED WINE.

Clarify with basic lead acetate and remove the excess of lead with sodium sulphate, as directed under the determination of sugar (p. 84). Filter, and treat a few drops of the filtrate as directed under (a).

22.—DETECTION OF PRESERVATIVES.

The preservatives to be tested for in wines are salicylic acid, benzoic acid, saccharin, abrastol, hydronaphthol, boric acid, borofluorids, and silicofluorids. Of these the salicylic and benzoic acids are both somewhat commonly employed. Abrastol is said to be used to some extent in Europe, but has not yet been reported in American wines. Hydronaphthol has been used in rare instances, and is still used with sufficient frequency to warrant more consideration than it usually receives from food laboratories. Boric acid is better known as a preservative for milk and meat preparations than for fruit and fruit preparations. It is sometimes used, however, in both wine and beer. Its detection is a somewhat more complicated matter than is the case with the other preservatives, because a small amount of boric acid is normal to wines. It is sometimes a difficult matter to fix the amount which may naturally occur. In order to make this test of practical value, therefore, it is essential that the determination of boric acid should be quantitative. The alkaline fluorids, as well as the alkaline borofluorids and silicofluorids, are coming into somewhat general use now as food preservatives, although they have not been frequently reported in wines.

^a Fruhauf and Ursic, Bericht u. die Versammlung Oesterreichischer Oenomiker in Bozen, 1886, p. 66; Borgmann, Anal. des Weines, 2d ed., p. 146.

^b Borgmann, Anal. des Weines, 2d ed., p. 143.

^c R. Fresenius, Ztschr. anal. Chem., 1890, 29, 20, 143 and 413; 1891, 30, 18, 452 and 583; 1893, 32, 189 and 312.

^d Egger, Arch. Hyg., 2, 373.

(a) SALICYLIC ACID.

Treat about 75 cc of wine with sufficient basic lead acetate^a to clarify, and filter through a ribbed filter paper. Add from 5 to 10 cc of dilute sulphuric acid (1-3), allow the precipitated lead sulphate to subside, and decant about 50 cc of the supernatant liquid into a separatory funnel. Extract with ether or chloroform and test for salicylic acid as directed under Food preservatives (p. 108). The writer has obtained much more satisfactory results by extracting after clarification as directed above, than by extracting the wine directly with a mixture of ether and petroleum ether, or by extracting the evaporated residue from the ether extract with petroleum ether. In no case should the volume of wine extracted for the detection of salicylic acid greatly exceed 50 cc. A similar reaction (with ferric chlorid) is said to be obtained sometimes from wines which contain no salicylic acid when a large volume of the wine is employed.^b

(b) BENZOIC ACID.

Acidify about 100 cc of wine with dilute (1-3) sulphuric acid, extract with ether and detect by Mohler's method, as described under Food preservatives (p. 109).

The presence of benzoic acid may be confirmed by neutralizing the aqueous solution of the extracted benzoic acid with sodium hydroxid, evaporating to a very small volume, and acidifying with sulphuric acid, when the presence of a large amount of benzoic acid is indicated by the formation of a white flocculent precipitate. The concentrated solution of the sodium salt may be further tested by adding a few drops of phenolphthalein solution, and then a very dilute solution of sodium hydroxid drop by drop, till an alkaline reaction is obtained, and a drop of a 0.5 per cent ferric chlorid solution, which should decolorize the phenolphthalein, when ferric benzoate is precipitated. The appearance of ferric benzoate is markedly different from that of ferric hydroxid, in that it is almost white when viewed by transmitted light and brown by reflected light, whereas ferric hydroxid has a brown color in both cases.

(c) DETECTION OF SACCHARIN.

Proceed as directed on page 109.

(d) SUCROL OR DULCIN.

(1) *Morpurgo's method.*^c

Evaporate about 100 cc of wine to a sirupy consistency after the addition of about 5 grams of lead carbonate, and extract the residue several times with alcohol of about 90 per cent; evaporate the alcoholic extract to dryness; extract the residue with ether, and allow the ether to evaporate spontaneously in a porcelain dish. Now add 2 or 3 drops each of phenol and concentrated sulphuric acid and heat for about five minutes on the water bath; cool; transfer to a test tube and pour ammonia or sodium hydroxid over the surface with the least possible mixing. The presence of dulcin is indicated by the formation of a blue zone at the plane of contact.

(2) *Jorisson's method.*^d

Suspend the residue from the ether extract obtained as directed above in about 5 cc of water; add from 2 to 4 cc of an approximately 10 per cent solution of mercuric nitrate, and heat from 5 to 10 minutes on the water bath. In the presence of sucrol, a violet blue color is formed, which is changed to a deep violet by the addition of lead peroxid.

^a See footnote on page 84.

^b Medicus, Ztschr. anal. Chem., 1896, 35, 398.

^c Ztschr. anal. Chem., 1896, 35, 104.

^d Ib., 628.

(e) DETERMINATION OF TOTAL SULPHUROUS ACID.

Distill 100 cc of wine in a current of carbon dioxid, after the addition of about 5 cc of a 20 per cent solution of glacial phosphoric acid, until 50 cc have passed over. Collect the distillate in a decinormal iodine solution in a flask closed with a stopper perforated with two holes, through one of which the end of the condenser passes and through the other a U-tube containing a portion of the standardized iodine solution. Twenty-five cc of decinormal iodine solution may be employed, diluted with water to give the desired volume. The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxid, adding 10 cc of phosphoric acid instead of 5 cc, and dropping into the distilling flask a piece of sodium bicarbonate weighing not more than a gram immediately before attaching to the condenser. The carbon dioxid liberated is not sufficient to expel the air entirely from the apparatus, but will prevent oxidation to a large extent. The U-tube trap may also be omitted if the end of the condenser tube be made to extend below the surface of the iodine solution, and the distillation conducted with a steady flame. When the distillation is finished, wash the contents of the U-tube into the flask and determine the excess of iodine with standardized thiosulphate solution. On account of its lack of permanence, the iodine solution employed should be titrated from time to time with a decinormal thiosulphate solution (containing 24.8 grams $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ per liter). The number of cubic centimeters of decinormal iodine solution employed, less the number of cubic centimeters of thiosulphate solution required at the end of the determination, is multiplied by 0.0032 for the grams of sulphur dioxide per 100 cc of wine.

Fairly accurate results may also be obtained by the following method:

Place 25 cc of a solution of potassium hydroxid containing 56 grams per liter in a flask of approximately 200-cc capacity. Introduce 50 cc of wine by means of a pipette, mix with the potassium hydroxid, and allow the mixture to stand for fifteen minutes with occasional agitation. Add 10 cc of 1-3 sulphuric acid and a few cubic centimeters of starch solution, and titrate the mixture with a N/50 iodine solution. Introduce the iodine solution as rapidly as possible and continue the addition until the blue color will last for several minutes. One cubic centimeter of N/50 iodine solution is equivalent to 0.00064 gram of sulphur dioxide. The number of cubic centimeters of the iodine solution employed, multiplied by 0.00128, gives the weight of the total sulphur dioxide expressed in grams per 100 cc.

(f) DETERMINATION OF FREE SULPHUROUS ACID.

Treat 50 cc of wine in a flask, having a capacity of approximately 200 cc, with about 5 cc of 1-3 sulphuric acid, add a small piece of sodium carbonate (about 0.5 gram) to expel the air, and titrate the sulphurous acid with N/50 iodine solution, as directed under total sulphurous acid.

One cubic centimeter of N/50 iodine solution is equivalent to 0.00064 gram of sulphur dioxide.

The number of cubic centimeters of iodine solution employed, multiplied by 0.00128, gives the weight of the free sulphurous acid expressed as sulphur dioxide in grams per 100 cc.

(g) DETECTION OF BETA-NAPHTHOL.

Extract 200 cc of wine with 10 cc of chloroform in a separatory funnel, add a few drops of alcoholic potash to the chloroform extract in a test tube, and place in a boiling water bath for two minutes. The presence of beta-naphthol is indicated by the formation of a deep blue color, which changes through green to yellow.

(h) DETECTION OF ABRASTOL.

(1) *Sinibaldi's method*.^a

Make 50 cc of the sample alkaline with a few drops of ammonia and extract with 10 cc of amyl alcohol (ethyl alcohol is added if an emulsion be formed). Decant the amyl alcohol, filter if turbid, and evaporate to dryness. Add to the residue 2 cc of a mixture of equal parts of strong nitric acid and water, heat on the water bath until half of the water is evaporated, and transfer to a test tube with the addition of 1 cc of water. Add about 0.2 gram of ferrous sulphate and an excess of ammonia, drop by drop, with constant shaking. If the resultant precipitate be of a reddish color, dissolve it in a few drops of sulphuric acid, and add ferrous sulphate and ammonia as before. As soon as a dark-colored or greenish precipitate has been obtained, introduce 5 cc of alcohol, dissolve the precipitate in sulphuric acid, and shake the fluid well and filter. In the absence of abrastol this method gives a colorless or light-yellow liquid, while a red color is produced in the presence of 0.01 gram of abrastol.

(2) *Sanglé-Ferrière's method*.^b

Boil 200 cc of wine with 8 cc of concentrated hydrochloric acid for one hour in a flask with reflux condenser attached. Abrastol is thus converted into beta-naphthol and is detected as directed under (g).

(i) BORIC ACID.

Boric acid is a normal constituent of wine and its qualitative detection in wine is therefore of little value unless a very heavy reaction is obtained. For methods of detection and estimation, see page 110.

(j) DETECTION OF FLUORIDS.

(1) *First method*.^c

Heat to boiling about 100 grams of wine, made slightly alkaline with ammonium carbonate and precipitate the fluorin with 2 or 3 cc of an approximately 10 per cent solution of calcium chlorid. Continue the boiling for five minutes, separate the precipitate by filtration, wash with a little water, dry, and ignite in a platinum crucible. Add 1 cc of strong sulphuric acid, cover the crucible with a watch glass coated with paraffin or wax, with a character marked through the wax so as to permit the watch glass to be etched at some point, and heat on a water bath for an hour at a temperature of from 75° to 80° C. One milligram can be readily detected by this method. The delicacy of the method is impaired by the presence of a small amount of silica in the ash of the wine.

(2) *Second method*.

If it is desired, the preceding method may be varied by mixing a small amount of precipitated silica with the precipitated calcium fluorid and placing it in a crucible covered by a watch glass which is not coated with paraffin, and to which a drop of water is suspended on the underside. Add 1 cc of concentrated sulphuric acid to the crucible, and heat for an hour at the temperature of 70° or 80° C. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica, while a ring is frequently etched at the circumference of the drop of water. Any fluosilicates and fluoborates present will also be indicated by this reaction.

^a Mon. Sci., 1893 [4], 7, 842.

^c Neviere and Hubert, Mon. Sci., 1895 [4], 9, 324.

^b Comp. rend. 1893, 117, 93.

(k) DETECTION OF FLUOBORATES AND FLUOSILICATES.

Make about 200 cc of wine alkaline with linewater, evaporate to dryness, and incinerate. Extract the crude ash first obtained with water, to which sufficient acetic acid has been added to decompose carbonates, filter, burn the insoluble portion, extract with dilute acetic acid, and again filter. The insoluble portion now contains calcium silicate and fluorid, while the filtrate will contain all the boric acid present.

(1) *First method.*^a

Incinerate the filter containing the insoluble portion, mix with a little precipitated silica, and place, with the addition of 1 or 2 cc of concentrated sulphuric acid, in a short test tube which is attached to a small U-tube containing a few drops of water. The test tube is now placed in a beaker of water, which is kept hot on the steam bath for from 30 to 40 minutes. If any fluorid be present the silicon fluorid generated will be decomposed by the water in the U-tube and will form a gelatinous deposit on the walls of the tube.

The filtrate is now tested as directed under boric acid. If both hydrofluoric and boric acids be present, it is probable that they were combined as borofluorid. If, however, silicon fluorid be detected and not boric acid, the operation is repeated without the introduction of the silica, in which case the formation of the silicon skeleton is conclusive of the presence of fluosilicate.^b

(2) *Second method.*

Incinerate the filter containing the insoluble portion in a platinum crucible, mix with a little precipitated silica, and add 1 cc of concentrated sulphuric acid. Cover the crucible with a watch glass to whose underside a drop of water is suspended, and heat an hour at the temperature of 70° or 80° C. The silicon fluorid which is formed is decomposed by the water, leaving a gelatinous deposit of silica. Test the filtrate for boric acid as described above.

(B) BEER.

1.—PREPARATION OF SAMPLE.

Transfer the contents of bottle or bottles into a large flask and shake vigorously to hasten the escape of carbon dioxide. The beer may then be poured into a second receptacle from under the foam.

2.—DETERMINATION OF SPECIFIC GRAVITY.

Follow the directions given for the determination of specific gravity in wine (p. 82).

3.—DETERMINATION OF ALCOHOL.

Follow the directions given for the determination of alcohol in wine (p. 82).

4.—DETERMINATION OF EXTRACT.

Ascertain the extract content corresponding to the specific gravity of the dealcoholized beer according to Table III.

For this purpose employ the formula:

$$sp = g + (1 - a)$$

^a Nevier and Hubert, Mon. Sci., 1895 [4], 9, 324.

^b It must be remembered that in an ash that contains an appreciable amount of silica, sulphuric acid will liberate silicon fluorid rather than hydrofluoric acid. The presence of a fluosilicate is indicated, therefore, and not of a fluorid.

in which sp is the specific gravity of the dealcoholized beer, g the specific gravity of the beer, and a the specific gravity of the distillate obtained in the determination of alcohol. In place of this formula, the residue from the distillation of alcohol is sometimes diluted to the original volume, and its specific gravity taken. This is often impracticable owing to the necessity of employing tannin to prevent foaming in the distilling flask, and owing to the coagulation of proteids during the distillation.

The extract of beer can not be accurately determined by evaporation and drying at the boiling point of water because of the dehydration of the maltose.

5.—DETERMINATION OF ORIGINAL GRAVITY OF WORT.

The various methods employed to obtain this figure depend on the fact that the sugars yield about half their weight of alcohol when fermented.

Employ the formula:

$$G = sp + si$$

in which G is the specific gravity of the original wort, sp the specific gravity of the dealcoholized beer (see 4), and si the amount of saccharine matter destroyed by fermentation—obtained from the following table:

Saccharine matter lost by fermentation.^a

1— a	0	1	2	3	4	5	6	7	8	9
0.000		0.0003	0.0006	0.0009	0.0012	0.0015	0.0018	0.0021	0.0024	0.0027
.001	0.0030	.0033	.0037	.0041	.0044	.0048	.0051	.0055	.0059	.0062
.002	.0066	.0070	.0074	.0078	.0082	.0086	.0090	.0094	.0098	.0102
.003	.0107	.0111	.0115	.0120	.0124	.0129	.0133	.0138	.0142	.0147
.004	.0151	.0155	.0160	.0164	.0168	.0173	.0177	.0182	.0186	.0191
.005	.0195	.0199	.0204	.0209	.0213	.0218	.0222	.0227	.0231	.0236
.006	.0241	.0245	.0250	.0255	.0260	.0264	.0269	.0274	.0278	.0283
.007	.0288	.0292	.0297	.0302	.0307	.0312	.0317	.0322	.0327	.0332
.008	.0337	.0343	.0348	.0354	.0359	.0365	.0370	.0375	.0380	.0386
.009	.0391	.0397	.0402	.0407	.0412	.0417	.0422	.0427	.0432	.0437
.010	.0442	.0447	.0451	.0456	.0460	.0465	.0476	.0475	.0480	.0485
.011	.0490	.0496	.0501	.0506	.0512	.0517	.0522	.0527	.0533	.0538
.012	.0543	.0549	.0554	.0559	.0564	.0569	.0574	.0579	.0584	.0589
.013	.0594	.0600	.0605	.0611	.0616	.0622	.0627	.0633	.0638	.0643
.014	.0648	.0654	.0659	.0665	.0671	.0676	.0682	.0687	.0693	.0699
.015	.0705	.0711	.0717	.0723	.0729	.0735	.0741	.0747	.0753	.0759

In this table, $1 - a$ is found by deducting from 1.0 the specific gravity of the alcohol distillate obtained in the determination of alcohol. In case of beer of high acidity, it must be increased by the value of b in the formula

$$b = 0.9 l - 0.14,$$

in which l is the percentage of acid calculated to lactic acid. The figure 0.14 is taken as the alcoholic equivalent of the average acid content of beer (ordinarily about 0.15 per cent lactic acid), and is deducted for that reason. The table here given is that of Graham, Hofmann, and Redwood,^b except that it is expressed here as

^a Allen, Com. Org. Anal., Vol. I.

^b Report on Original Gravities, 1852; Allen, Commercial Organic Analysis, 3d edition, Vol. I, p. 136.

specific gravity instead of parts per thousand. It has been adopted by the English excise.

The method outlined above was employed by the same authors, except that they determined the specific gravity of the dealcoholized beer, directly on the residue from the alcohol determination after diluting to the volume of beer employed.

6.—DETERMINATION OF THE DEGREE OF FERMENTATION.

Calculate from the formula

$$D = \frac{100 \text{ } sp}{G}$$

in which D is the degree of fermentation, sp the specific gravity of the dealcoholized beer, and G the gravity of the original wort.

7.—DETERMINATION OF TOTAL ACIDS.

Heat 20 cc of the sample to incipient boiling to liberate carbon dioxid, and titrate with decinormal sodium hydroxid, using neutral litmus paper as indicator. Each cubic centimeter of decinormal alkali employed is equivalent to 0.009 grams of lactic acid. The number of cubic centimeters of decinormal alkali employed in titrating 20 cc of beer is multiplied by 0.045 for the acidity expressed as grams of lactic acid per 100 cc.

8.—DETERMINATION OF VOLATILE ACIDS.

Follow the directions given for the determination of volatile acids in wine (p. 84). This determination is rarely of value in sound beer.

9.—DETERMINATION OF REDUCING SUGAR.

Proceed as directed on page 85, but boil four minutes instead of two. Express the result in terms of maltose equivalent to copper reduced, according to Table IX.

10.—DETERMINATION OF DEXTRIN.

Deduct from the extract the sum of the maltose, protein, glycerol, total acids, and ash. While this method is only approximate, it is sufficiently accurate for most purposes.

If a more accurate determination is desired, 50 cc may be treated by Sachsse's method for the hydrolization of starch, and dextrose determined by copper reduction, according to Allihn's method. From the amount of dextrose so found, 95 per cent of the amount of maltose present in the beer is deducted (20 parts maltose are equivalent to 19 parts dextrose) and the remainder multiplied by 0.9.

11.—DETERMINATION OF GLYCEROL.

Proceed as directed on page 82. The milk of lime is added during evaporation after the carbon dioxid has been expelled.

12.—DETERMINATION OF ASH.

Evaporate 25 cc to dryness, ignite as directed on page 83, and weigh.

13.—DETERMINATION OF PHOSPHORIC ACID.

Employ the official gravimetric or volumetric method,^a using the residue obtained in the determination of ash.

14.—DETERMINATION OF PROTEIN.

Employ the Kjeldahl or the Gunning method for the determination of nitrogen, and multiply the result by 6.25.

^aU. S. Dept. of Agr., Div. of Chem., Bul. 46, p. 13.

15.—DETERMINATION OF CARBON DIOXID.

(a) BOTTLED GOODS.

Pierce the cork with a champagne tap.^a Connect with a suitable absorption apparatus, placing an Erlenmeyer flask between the bottle and absorption tubes to allow the bubbles to break and prevent them from passing beyond it. The accompanying illustration (fig. 3) of an apparatus devised by Crampton and Trescot^b answers admirably for this purpose. Immerse the bottle in water in a suitable vessel—such as an ether can with the top cut away, as shown in the cut—allow the gas to escape slowly, and when it ceases to flow spontaneously heat gradually to about 80° C. and maintain this temperature for about half an hour, shaking the bottle from time to time. Then disconnect the bottle, replace it with a soda-lime tube and draw a current of air through the apparatus. The increase in weight of the absorption tube gives the amount of carbon dioxide. The volume of beer employed is also weighed or measured.

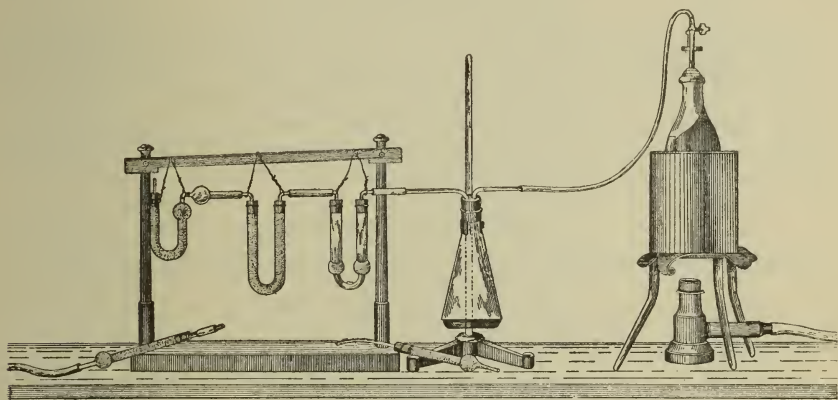


FIG 3.—Apparatus for the determination of carbon dioxide.

When the bottle containing the sample is closed with a patent stopper, the latter may sometimes be replaced by a rubber stopper fitted with stopcock tube. Where the pressure is so great that this is not practicable, such samples may be treated as directed under "Bulk Goods."

(b) BULK GOODS.

Close a round-bottom flask of about 700-cc capacity with a two-hole rubber stopper fitted with two stopcock tubes bent at right angles—one passing to the bottom of the flask and the other ending just below the stopper.^c Produce a partial vacuum in the flask by means of an aspirator, and weigh the flask. Dip the end of one of the stopcock tubes below the surface of the beer, or, better, attach it by means of a rubber tube to a champagne tap or small faucet screwed into the cask, and allow about 300 cc of the sample to enter the flask. Weigh the flask and contents, and proceed as directed under "Bottled Goods." Somewhat better results may be

^a Hassall, Food Adulteration S. & C. Used by Wiley (Am. Chem. Jour., 1886, 8, 200) in the examination of koumiss, and by Crampton in the examination of beer. Crampton found it necessary to regrind the cocks and ream off the thread, leaving a smooth tube. U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 3, p. 294.

^b U. S. Dept. of Agr., Div. of Chem., Bul. 13, pt. 3, p. 293.

^c Windisch (Das chemische Laboratorium des Brauers, p. 247), employs ordinary glass tubes provided with rubber tubing and screw cocks.

obtained by placing a reflux condenser between the flask and absorption apparatus, and heating the flask over a burner to the boiling point. Attach the other stopcock tube to a soda-lime guard tube and pass a current of air through the apparatus. The amount of carbon dioxide is ascertained by the increase in weight of the absorption tube.

16.—DETECTION OF PRESERVATIVES.

Proceed as directed on pages 88 and 107.

(C) DISTILLED LIQUORS.

1.—DETERMINATION OF SPECIFIC GRAVITY.

Proceed as directed on page 82. Owing to the high alcohol content of distilled liquors, care must be exercised that the temperature at which specific gravity is determined be as nearly 15.6°C. as possible.

2.—DETERMINATION OF ALCOHOL.

Measure 50 cc of the sample (at 15.6°C.) into a distilling flask, dilute with 100 cc of water, and proceed as directed on page 82. The sample of distilled liquor taken for the determination of alcohol is diluted more than in the case of wine because of the errors attending distillates high in alcohol, errors due to evaporation and to making up to volume at temperatures varying slightly from 15.6°C. All measurements must be made at about that temperature.

3.—DETERMINATION OF EXTRACT.

Evaporate 100 cc to sirupy consistency and proceed as directed on page 83.

4.—DETERMINATION OF ASH.

Proceed as directed on page 83.

5.—DETERMINATION OF ACIDITY.

Titrate 100 cc with decinormal sodium hydroxid using phenolphthalein as indicator. The number of cubic centimeters employed is multiplied by 0.0060 for the acidity expressed in grams of acetic acid per 100 cc.

6.—DETERMINATION OF SUGAR.

Proceed as directed on page 85.

7.—DETERMINATION OF FUSEL OIL.^a

The apparatus recommended for this determination is Bromwell's modification of Roese's fusel-oil apparatus. (See fig. 4.)

The reagents required are fusel-free alcohol that has been prepared by fractional distillation over caustic soda or caustic potash, rejecting the first one-fifth and the last three-fifths of the distillate, and diluted to exactly 30 per cent by volume (sp. gr. 0.96541 at 15.6°C.), chloroform, freed from water and redistilled, and sulphuric acid (sp. gr. 1.2857 at 15°C.).

Distill slowly 200 cc of the sample under examination till about 175 cc have passed over, allow the distilling flask to cool, add 25 cc of water, and distill again till the total distillate measures 200 cc. Dilute the distillate to exactly 30 per cent by volume^b (sp. gr. 0.96541 at 15.6°C.).

^a Windisch, Arb. kais. Gesamt., Vol. V, p. 390.

^b The following is an accurate method for diluting any given alcohol solution to a weaker solution of definite percentage: Designate the volume percentage of the stronger alcohol by V and that of the

Now prepare a water bath, the contents of which are kept at exactly $15^{\circ}\text{C}.$, and place in it the apparatus (covering the end of the tube with a rubber cap to prevent wetting the inside of the tube) and flasks containing the 30 per cent fusel-free alcohol, chloroform, sulphuric acid, and the distillate diluted to 30 per cent by volume. When the solutions have all attained the temperature of $15^{\circ}\text{C}.$, fill the apparatus to the 20-cc mark with the chloroform, drawing it through the lower tube by means of suction, add 100 cc of the 30 per cent fusel-free alcohol and 1 cc of the sulphuric acid, invert the apparatus and shake vigorously for two or three minutes, interrupting once or twice to open the stopcock for the purpose of equalizing pressure. Allow the apparatus to stand for one hour in water that is kept at the temperature^a of $15^{\circ}\text{C}.$, turning occasionally to hasten the settling of the chloroform and note the volume of the chloroform. After thoroughly cleansing and drying the apparatus repeat this operation, using the diluted distillate from the sample under examination in place of the fusel-free alcohol. The increase in the chloroform volume with the sample under examination over that with the fusel-free alcohol is due to fusel oil, and this difference (expressed in cubic centimeters) multiplied by the factor 0.663 gives the volume of fusel oil in 100 cc, which is equal to the percentage of fusel oil by volume in the 30 per cent distillate. This must be calculated to the percentage of fusel oil by volume in the original liquor.

Example.—A sample of liquor contains 50 per cent of alcohol by volume. The increase in the chloroform volume with the 30 per cent fusel-free alcohol is 1.42 cc. The increase in the chloroform volume with the distillate from the liquor under examination diluted to 30 per cent is 1.62 cc; difference, 0.20 cc. The volume of fusel oil in 100 cc of the 30 per cent distillate, then, is $0.20 \times 0.663 = 0.1326$, and by the proportion 30:50::0.1326:0.221 we obtain the percentage of fusel oil by volume in the original liquor.

8.—DETERMINATION OF ALDEHYDES.^b

Dissolve 0.5 grams of fuchsin in about 100 cc of water; add a solution containing the same weight of sulphurous acid (H_2SO_3); dilute to a liter and filter. With 1 volume of this reagent mix 2 volumes of the 30 per cent distillate obtained in the determination



FIG. 4.—Bromwell's fusel-oil apparatus.

weaker alcohol by v. Mix v volumes of the stronger alcohol with water to make V volumes of the product. Allow the mixture to stand till full contraction has taken place and till it has reached the temperature of the original alcohol and water and make up any deficiency in the V volumes with water.

Example.—It is desired to dilute a distillate containing 50 per cent of alcohol by volume until it contains 30 per cent. To 30 volumes of the 50 per cent alcohol add enough water to make 50 volumes, or place 150 cc of the distillate in a 250-cc flask, fill to the mark with water, mix, cool, and fill to the mark again.

Owing to the extreme difficulty of preparing distillates of exactly 30 per cent, slight variations may be corrected by increasing or decreasing the chloroform reading, as suggested by Sell, 0.003 cc for each 0.01 per cent variation in strength of alcohol from 30 per cent. Such variation, however, should not exceed 0.02 per cent.

^aThe temperature must be held as nearly $15^{\circ}\text{C}.$ as possible. If any variations occur the chloroform must be increased or decreased 0.046 cc for every degree above or below that temperature (Gebek & Stutzer, *Ztschr. ang. Chem.*, 1893, 132).

^b *Medicus Forsch. uber Lebensm.*, 1895, 2, 299.

of fusel oil. Treat in the same manner and at the same time a solution of 30 per cent (by volume) aldehyde-free alcohol containing 0.05 grams of acetic aldehyde per liter. After two minutes, match the colors of the two mixtures by dilution of the stronger with 30 per cent aldehyde-free alcohol, or by means of a colorimeter, and express the result as acetic aldehyde.

9.—DETERMINATION OF ETHEREAL SALTS.

After the determination of the volatile acids the neutralized distillate is transferred to a flask connected with a reflux condenser, treated with 25 cc of tenth normal sodium hydroxid, and boiled one-half hour. The flask and contents are then cooled, 25 cc of decinormal hydrochloric acid added, and the excess of acid titrated with sodium hydroxid, using phenolphthalein as indicator. The number of cubic centimeters of decinormal alkali used in this titration, multiplied by 0.0088, is equal to the weight in grams of ethereal salts (calculated as ethyl acetate) in the volume of liquor taken for the determination.

10.—DETERMINATION OF FURFUROL.^a

Treat 5 cc with 5 drops of colorless anilin and 8 drops of acetic acid. After fifteen minutes, compare colorometrically with 5 cc of a solution containing 0.05 grams of furfural per liter which has been subjected to the same treatment.

11.—DETERMINATION OF COLORING MATTER.

Proceed as directed under methods for the detection of coloring matter, (p. 111). For the detection of caramel use the method of Crampton and Simons,^b which depends on the insolubility of caramel in ether. Evaporate 50 cc of the sample nearly to dryness on the water bath, wash into a 50-cc flask, add 25 cc of absolute alcohol, cool to a definite temperature, and dilute to mark with water. Transfer 25 cc to an apparatus of the general description of Bromwell's fusel-oil apparatus (page 97), but graduated so that the lower bulb holds 25 cc to a definite mark on the stem, which may be of larger tare than in Bromwell's apparatus. Add 50 cc of ether and shake at intervals for half an hour, let settle, and siphon water through the lower tube until the aqueous layer reaches the 25-cc mark. Mix the whole, remove the aqueous layer, and compare by means of a tintometer with the 25 cc of the solution which were not treated with ether. Express the amount of color removed on the percentage basis.

XV.—BAKING POWDERS AND BAKING-POWDER CHEMICALS.

By A. L. WINTON,

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All the processes hereafter described, except determination of acidity, may be employed in the analysis of baking powders, and all the processes, except determination of carbonic acid, in the analysis of cream of tartar and its substitutes. The sample under examination is entirely removed from the package, carefully mixed, and passed through a sieve without grinding.

1.—DETERMINATION OF TOTAL CARBON DIOXID.^c

This determination is made by the absorption method, and any apparatus may be employed which gives accurate results when checked with pure calcite. Whatever apparatus is chosen the tubes and materials used for absorbing and drying the carbon dioxide may be varied according to the preference of the analyst. Those

^a Windisch. *Forsch. über Lebens.*, 1897, **4**, 369.

^b *Jour. Am. Chem. Soc.*, 1890, **22**, 810.

^c See Appendix, p. 156.

mentioned below are selected because the details have been carefully worked out by the originators.

According to the amount of absorbent employed the weight of sodium carbonate or calcium carbonate may vary from 0.25 to 1.00 gram, and about twice as much baking powder may be used. The corrections for temperature and pressure given with the Heidenhain apparatus may ordinarily be disregarded.

(a) KNORR'S APPARATUS.

(1) *Description of apparatus.*

This apparatus (fig. 5) employs only ground-glass joints, and may be quickly made ready for use or taken to pieces and packed away. On the other hand, it is inflexible

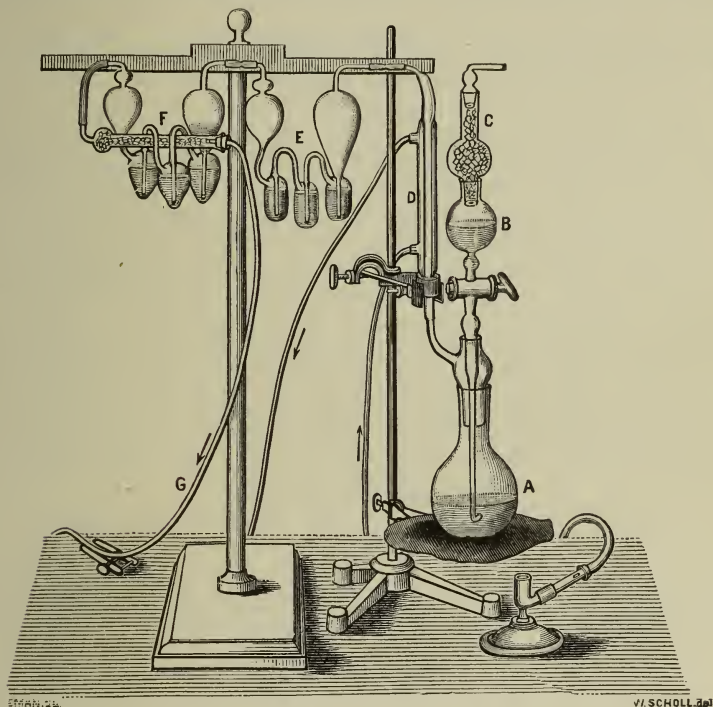


FIG. 5.—Knorr's apparatus for the determination of carbon dioxide: A, Distilling flask fitted to condenser by a ground-glass stopper. B, Reservoir containing acid. C, Soda-lime tube fitted to acid reservoir by a ground-glass stopper. D, Condenser. E, Liebig bulb filled with sulphuric acid. F, Liebig bulb filled with a solution of potassium hydroxid for the absorption of carbon dioxide and followed by a calcium-chlorid tube. An additional guard tube filled with soda lime should follow the tube F, though not shown in the cut.

and must be carefully handled, and has the additional disadvantage that broken parts can not readily be replaced. Therefore it is of more value for occasional determinations than for a long series.^a

^aThe small calcium chlorid tube shown in the cut attached to the potash bulb F is usually replaced by a second Liebig bulb filled with sulphuric acid. Better results are obtained if the same drying tubes are used before and after the potash bulb. Many analysts prefer to replace the bulb F and attached calcium chlorid tube by two U-tubes filled with sifted soda lime. When the second tube shows a material increase in weight it is placed first, and the first tube refilled and placed in the second position.

(2) *Materials.*

The potassium hydroxid solution usually employed for absorbing carbon dioxid has a specific gravity of about 1.27. Many analysts, however, prefer a solution having a specific gravity of 1.55.

The calcium chlorid and soda lime employed should be finely granulated and freed from dust with a sieve.

(3) *Manipulation.*

The quantity of baking powder to be examined is placed in a distilling flask, which must be perfectly dry.^a The flask is closed with a stopper carrying the tube connecting with the absorption apparatus and also with the funnel tube. The tubes in which the carbon dioxid is to be absorbed are weighed and attached to the apparatus. In case two Liebig bulbs are employed, one for potassium hydroxid and the other for sulphuric acid, to absorb the moisture given up by the potassium hydroxid solution, it will be necessary to weigh them separately. If two soda-lime tubes are employed it will be found advantageous to weigh them separately and fill the first tube anew when the second tube begins to increase in weight materially. The tube B is nearly filled with hydrochloric acid (sp. gr. 1.1), and the guard tube C placed in position. The aspirator is now started at such a rate that the air passes through the Liebig bulbs at the rate of about two bubbles per second. The stopper of the funnel tube is opened and the acid allowed to run slowly into the flask, care being taken that the evolution of gas shall be so gradual as not to materially increase the current through the Liebig bulb. After the acid has all been introduced, the aspiration is continued, when the contents of the flask are gradually heated to boiling, the bulb in tube B being closed. While the flask is being heated the aspirator tube may be removed, although many analysts prefer when using ground-glass joints to aspirate during the entire operation. The boiling is continued for a few minutes after the water has begun to condense in D, when the flame is removed, the valve in the tube B opened, and the apparatus allowed to cool with continued aspiration. The absorption tubes are then removed and weighed, the increase in weight being due to carbon dioxid.

(b) HEIDENHAIN'S APPARATUS.

(1) *The apparatus.*

This was originated by G. J. Mulder and recommended and improved by Kolbe, Stolba, and Fresenius,^b and has been modified by H. Heidenhain,^c as shown in Fig. 6, which is drawn on a scale of 1 : 12. It consists of—

- A. A cylinder filled with soda-lime to free the air from carbon dioxid. A thick layer of cotton prevents soda-lime dust from being carried over.
- B. Glass cock to regulate the air current, which finds resistance at C.
- C. A capillary contraction.
- D. Funnel tube of peculiar shape. The funnel is cylindrical, three-fourths of an inch wide and 4 inches long, and is reduced to half its width at the bottom, so as to make a neck for a perforated rubber stopper into which—
- E. A glass tube is tightly fitted, allowing the stopper to be taken out and put in by the glass tube.
- F. Evolution flask, ordinarily of 150-cc capacity, for foaming liquids of 300-cc capacity.
- G. Return condenser, simply a glass tube of one-fourth of an inch bore, around which a small lead pipe is wound. The tube following the condenser contains a few pieces of calcium chlorid, to retain the bulk of the moisture. It is refilled when contents are liquefied.

^a See Appendix, p. 157.

^b Quant. Anal., vol. 1, p. 449, and vol. 2, p. 308, German edition.

^c Jour. Am. Chem. Soc., 1896, 18, 1.

- H. U-tube filled with coarse calcium chlorid.
- K. Filled at I with a 3-inch long column of pumice stone impregnated with copper sulphate completely dehydrated at 150° C. The rest is filled with fine calcium chlorid.
- L. Cock to close the apparatus when not in use.
- M. First absorption tube about one-half inch in diameter and 5 inches long, filled mainly with soda-lime, with a little calcium chlorid at the side at which the air current enters.
- N. Second absorption tube of same size as M, filled half with soda-lime and half with calcium chlorid. Place the side containing calcium chlorid toward the end of the apparatus where the air current leaves.
- O. Guard-tube containing calcium chlorid toward N and soda-lime toward P.
- P. Indicator tube trapped with glycerin.
- R. Safety bottle to receive water which may be sucked back from—
- S. The aspirator, which is a Mariotte's bottle of about 4 liters capacity.

(2) *Materials.*

Use calcium chlorid dehydrated at 200° C, not fused. Grind it coarsely in a coffee mill and sift through No. 18 wire gauze to remove the extremely coarse, and through No. 30 wire gauze to remove the very fine. Prepare a large quantity of such calcium chlorid at the beginning and use this for the tubes K, M, and N. The reason for this is that the current of air must leave the weighed tubes with the same content of moisture as it entered them, which only can be attained if the absorbent in K and N is of the same nature and quality.

The soda-lime^a for the weighed tubes is ground and sifted in the same way. It should not be too dry, as it must not absorb moisture to a higher degree than calcium chlorid. The tubes M and N should hold about 20 grams filling each, making M's capacity for carbon dioxid almost 1 gram and N's capacity for moisture 0.2 gram. M should be refilled when its weight has increased 0.75 gram, and N after an increase of 0.1 gram in weight.

Use best rubber for all connections, applying a trace of castor oil as lubricator. For connections of the weighed tubes use rubber tubing boiled in weak lye, washed and dried. Apply also a little castor oil, which is thoroughly wiped off again before connecting the tubing.

Before using the apparatus fill H and K with carbon dioxid, in order to saturate the alkalinity of the calcium chlorid, and exhaust after several hours.

(3) *Manipulation.*

Weigh M and N, taking precaution that they are of the same temperature as the air in the balance-room. Shortly before weighing, open the tubes for a moment to allow equalization of air. Note thermometer and barometer. Connect tubes with the apparatus and make sure that all joints are tight by closing A at the bottom, opening all cocks, starting the aspirator and observing P, in which the liquid must soon come to a standstill. Then disconnect the aspirator, close B, remove F, put in the substance^b (use about 1 gram of sodium carbonate or calcium carbonate, or about 2 grams of baking powder), connect F, and start the cooler. Fill acid and water through D, lifting E slightly and allowing only small quantities of the acid and water to enter at the time. (Use only water made free from carbon dioxid by boiling.) Light the burner, heat to boiling, and reduce the flame to keep the liquid just at the boiling point. If no more air passes P, start the aspiration. When water stops running, open B carefully and adjust the outflow of the aspirator by raising or lowering the syphon to half the safe speed.

^a An excellent method for the preparation of soda-lime is given by Benedict and Turner, Jour. Amer. Chem. Soc., 1899, 21, 396.

^b See Appendix, p. 157.

(In order to find the allowable rapidity of the air current proceed as follows: Charge the apparatus exactly as for an analysis, leaving out the carbonate. Start to

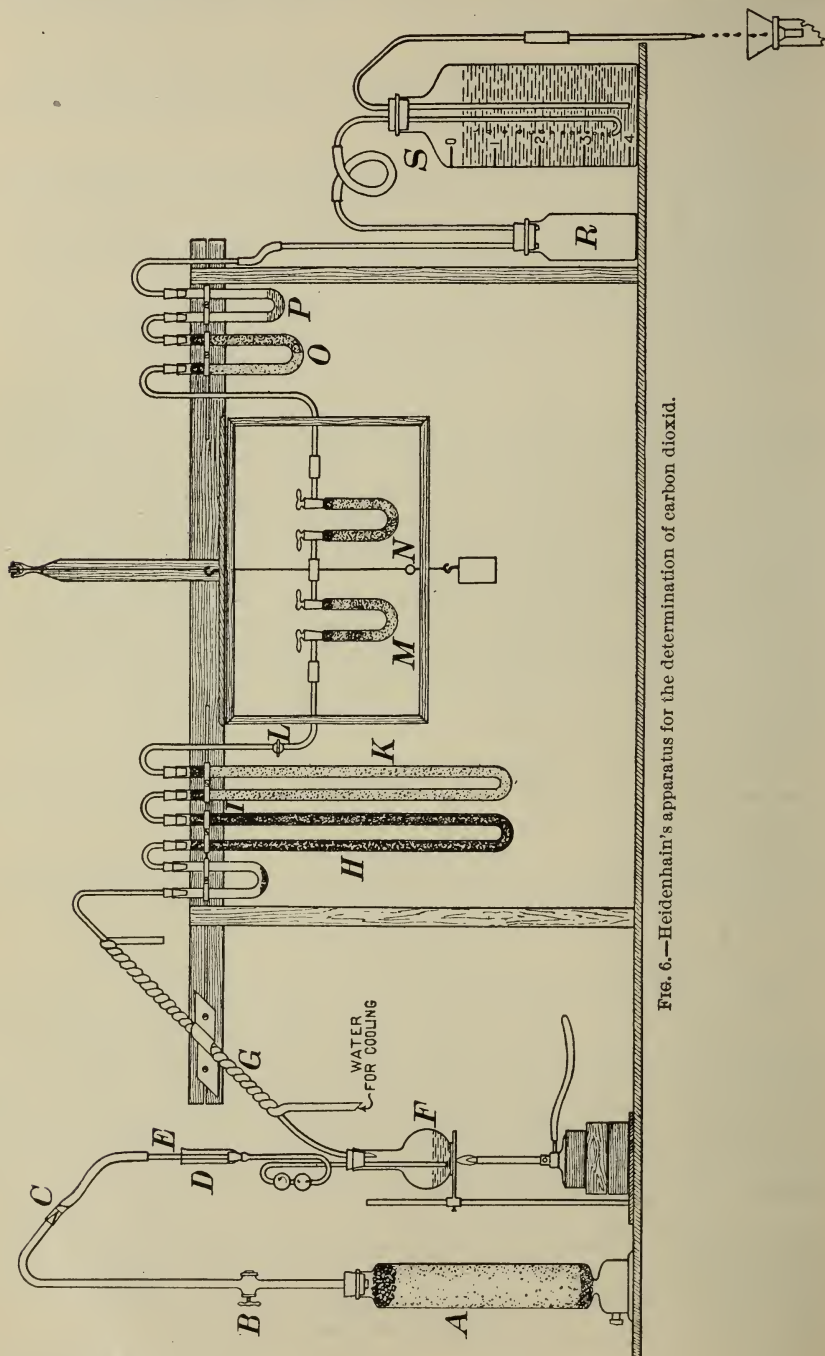


FIG. 6.—Heidenhain's apparatus for the determination of carbon dioxide.

aspirate at the rate of about 50 cc per minute. After two liters have been aspirated weigh the tubes. If they have lost in weight, repeat the experiment with 40 cc per

minute, and so on until the weight of the tubes remains constant. If the work has been done with due precaution, the first tube must have lost just as much as the second has gained. Do not exceed the safe speed thus found.)

After M has become cool increase the current to full safe speed and aspirate altogether 3 liters, continuing boiling to the end of aspiration. After the tubes have assumed the temperature of the balance-room open for a moment, weigh, note again thermometer and barometer, and apply correction according to the following formulæ:

$$-(A^2 - A^1) \times T \text{ and } + (B^2 - B^1) \times B$$

in which

A^1 is the temperature at first weighing in °C,

A^2 is the temperature at second weighing in °C,

B^1 is the air pressure at first weighing in mm,

B^2 is the air pressure at second weighing in mm,

and T and B are constants found as follows:

G=weight of empty tubes.

F=weight of fillings.

The specific gravity of glass =2.7.

The specific gravity of filling=2.0.

The specific gravity of brass =8.5.

Change of weight of 1 cc air with 1°=0.0000039 gram.

Change of weight of 1 cc air with 1 mm pressure=0.0000015 gram.

From above follows:

$$\text{Volume of tubes and fillings} = \frac{G}{2.7} + \frac{F}{2.0}$$

$$\text{Volume of brass weights} = \frac{G+F}{8.5}$$

and

$$\frac{G}{2.7} + \frac{F}{2.0} - \frac{G+F}{8.5} = V,$$

representing the differential volume affected by temperature and pressure and being a constant for the tubes.

Now,

$$T = V \times 0.0000039 \text{ gram, and}$$

$$B = V \times 0.0000015 \text{ gram.}$$

Observe that rise of temperature makes the air lighter, consequently the tubes heavier. Therefore the correction must be negative. On the other hand, increased pressure has the opposite effect, making the correction positive.

Example:

G = 80. F = 40, from which follows

V = 35.5, and

T = 0.00014 gram, and

B = 0.00005 gram.

Now, if $A^1 = 25^\circ$,

$A^2 = 27^\circ$,

$B^1 = 759 \text{ mm,}$

$B^2 = 756 \text{ mm,}$

then the correction for temperature will be—

$$- 0.00028 \text{ gram,}$$

and for air pressure—

$$- 0.00015 \text{ gram,}$$

making a total of—

$$- 0.00043 \text{ gram.}$$

2.—DETERMINATION OF RESIDUAL CARBON DIOXID.^a

Weigh 2 grams of baking powder into a flask suitable for the subsequent determination of carbonic acid, add 20 cc of cold water, and allow to stand 20 minutes. Place the flask in a metal drying cell surrounded by boiling water, and heat with occasional shaking for 20 minutes.

To complete the reaction and drive off the last traces of gas from the semisolid mass, heat quickly to boiling over a lamp, and boil for one minute. Aspirate until the air in the flask is thoroughly changed, and determine the residual carbon dioxide by absorption, as described under *total* carbonic acid.

The process described,^b based on the methods of McGill^c and Catlin,^d imitates as far as practicable the conditions encountered in baking, but in such a manner that concordant results may be readily obtained on the same sample, and comparable results on different samples.

3.—DETERMINATION OF AVAILABLE CARBON DIOXID.

Subtract the residual carbon dioxide from the total.*

4.—DETERMINATION OF ACIDITY.

(For cream of tartar and its substitutes.)

Dissolve one gram of the material in hot water and titrate with standard fifth-normal potassium hydroxid solution, using phenolphthalein as indicator.

5.—DETECTION OF TARTARIC ACID, FREE OR COMBINED.^e

Applicable in presence of phosphates.

Shake repeatedly about 5 grams of the sample with about 250 cc of cold water in a flask and allow the insoluble portion to subside. Decant the solution through a filter and evaporate the filtrate to dryness. To the dry powdered residue add a few drops of a 1 per cent solution of resorcin and about 3 cc of strong sulphuric acid. Heat slowly. A rose-red color indicates tartaric acid, the color being discharged on dilution with water.

6.—DETECTION OF FREE TARTARIC ACID.

Extract 5 grams of the powder with absolute alcohol and evaporate the alcohol from the extract. Dissolve the residue in dilute ammonia, transfer to a test tube, add a good-sized crystal of silver nitrate, and heat gently. Tartaric acid is indicated by the formation of a silver mirror. If desired, the absolute alcohol extract may be tested by the Wolff method, as described under 5.

7.—DETERMINATION OF TOTAL TARTARIC ACID.^f

The following is the Goldenberg-Geromont-Heidenhain method, applicable only in the absence of aluminum salts, calcium salts, and phosphates:

Into a shallow porcelain dish, 6 inches in diameter, weigh out 2 grams of the material and sufficient potassium carbonate to combine with all tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc of cold water and add 5 cc of 99 per cent acetic acid. Stir for half a minute with a glass rod bent near the end. Add 100 cc of 95 per cent alcohol, stir violently for 5 minutes and allow to settle at least 30 minutes. Filter on a Gooch crucible with a thin layer of paper pulp, and wash with 95 per cent alcohol until 2 cc of the filtrate do not change the

^a See Appendix, p. 157.

^b Conn. Agr. Exp. Sta. Rep., 1900, p. 169.

^c Lab. Inland Rev. Dept., Ottawa, Canada, Bul. 68, p. 31.

^d Baking powders. A Treatise on the Character, Methods for Determination of the Values, etc., p. 20.

^e Wolff, Rev. chim. anal. appl., 1899, 4, 263.

^f See Appendix, p. 158.

color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc of hot water and add standard fifth-normal potassium hydroxid solution, leaving it still strongly acid. Boil for one minute. Finish the titration, using phenolphthalein as indicator and correct the reading by adding 0.2 cc. One cubic centimeter of fifth-normal potassium hydroxid solution is equivalent to 0.026406-gram tartaric anhydrid ($C_4H_4O_5$), 0.03001 gram tartaric acid ($H_2C_4H_4O_6$), and 0.03763 gram potassium bitartrate ($KHC_4H_4O_6$).

The standard of the potassium hydroxid solution should be fixed by pure dry potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing no free tartaric acid, obtained by calculation from the tartaric acid, with those obtained by calculation from the potassium oxide.^a

8.—DETERMINATION OF STARCH.

(a) DIRECT INVERSION METHOD.

(For all baking powders and baking chemicals free from lime.)

Weigh 5 grams of the powder into a graduated 500 cc flask. Convert into dextrose by the Sachsse's method and determine the reducing power of the solution by the Allihn method, as described under Spices (p. 57).

(b) INDIRECT METHOD.^b

(For phosphate, alum phosphate, and all other baking powders containing lime.)

Mix 5 grams of the powder in a graduated 500-cc flask, with 200 cc of 3 per cent hydrochloric acid, and allow the mixture to stand for one hour, with frequent shaking. Filter on a Schleicher and Schuell No. 575 11 cm hardened filter, taking care that a clear filtrate is obtained. Rinse the flask once, without attempting to remove all the starch, and wash the paper twice with cold water. Carefully wash the starch from the paper back into the flask, with 200 cc of water, using a small wash bottle. Add 20 cc of 25 per cent hydrochloric acid and proceed according to Sachsse's method. Determine reducing power by Allihn's method.

The treatment with 3 per cent hydrochloric acid, without dissolving the starch, effectually removes the lime, which otherwise would precipitate as tartrate in the alkaline copper solution

(c) M'GILL METHOD.

The following modification of McGill's method is valuable for check purposes:

Digest one gram of the powder with 150 cc of 3 per cent hydrochloric acid for 24 hours at the room temperature, with occasional shaking. Filter on a Gooch crucible, wash thoroughly with cold water and finally once with alcohol and once with ether. Dry at 110° C. (4 hours is usually sufficient), cool and weigh. Burn off the starch and weigh again. To obtain the weight of starch subtract the weight after burning from the weight after drying at 110° C.

The results by this method on cream of tartar powders and tartaric acid powders agree closely with those obtained by copper reduction. On phosphate, alum, and alum-phosphate powders the results are usually satisfactory, but in some instances they may be over 2 per cent too high.

9.—DETERMINATION OF POTASSIUM BITARTRATE.

If, as is usually the case, no other potassium salt but the bitartrate is present, multiply the percentage of total potash determined as directed under 12, d, by 3.9936.

^a Conn. Agr. Exp. Sta. Rep., 1900, p. 180.

^b After Winton, Conn. Agr. Exp. Sta. Rep., 1900, p. 174.

10.—DETERMINATION OF FREE TARTARIC ACID.

Calculate the percentage of tartaric anhydrid combined with the potash as bitartrate (if any) and subtract this from the percentage of total tartaric anhydrid. The difference is the tartaric anhydrid originally added as the free acid, although if the sample has been kept for a long time or has been improperly stored, a portion or all of this acid may exist at the time of analysis as the sodium salt resulting from the reaction in the can with the sodium bicarbonate. Multiply by 1.1365 to obtain the percentage of tartaric acid.

11.—DETECTION OF ALUM IN PRESENCE OF PHOSPHATES. ^a

(a) IN BAKING POWDER.

Burn to an ash about 2 grams of the sample in a platinum dish. Extract with boiling water and filter. Add to the filtrate a few drops of ammonium chloride solution. A flocculent precipitate indicates alum.

(b) IN CREAM OF TARTAR.

Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash, and proceed as in (a).

12.—EXAMINATION OF ASH. ^b

(a) DETERMINATION OF INSOLUBLE ASH AND PREPARATION OF SOLUTIONS.

Char 5 grams of the material in a platinum dish at a heat below redness. Boil the carbonaceous mass with dilute hydrochloric acid, filter into a graduated 500-cc flask, and wash with hot water. Return the residue, together with the paper, to the platinum dish and burn to a white ash. Boil again with hydrochloric acid, filter, wash, unite the two filtrates, and dilute to 500 cc.

Incinerate the residue after the last filtration for the determination of ash insoluble in acid.

(b) IRON AND ALUMINA. ^c

Draw an aliquot portion of 100 cc and separate silica, if necessary. Mix the solution with sodium-phosphate solution in excess of what is required to form normal aluminum phosphate. Add ammonia until a precipitate remains on stirring, then hydrochloric acid drop by drop until the precipitate dissolves. Heat the solution to about 50° C., mix with a considerable excess of 50 per cent ammonium-acetate solution and 4 cc of 80 per cent acetic acid.

As soon as the precipitate of aluminum phosphate, mixed with a little iron phosphate, has settled, collect on a filter, wash with hot water, ignite, and weigh.

Fuse the mixed phosphates with ten parts of sodium carbonate, dissolve in dilute sulphuric acid, reduce with hydrogen sulphid and determine the iron by the volumetric permanganate method. In the same solution determine the phosphoric acid. To obtain the weight of Al_2O_3 , subtract the sum of the weights of Fe_2O_3 and P_2O_5 from the weight of the mixed phosphates.

(c) LIME.

Heat the filtrate from the mixed phosphates, which is acid with acetic acid, to 50° C. and precipitate with ammonium oxalate. Filter, wash, ignite over a Bunsen burner, and finally convert into oxid by heating over a blast lamp.

^aThirty-first An. Rep. Mass. State Board of Health, 1899, p. 638.

^bConn. Agr. Exp. St., Rep. 1900, p. 178.

^cSee Appendix, p. 160.

(d) POTASH AND SODA.^a

Evaporate an aliquot portion of the solution, prepared as described, nearly to dryness to remove the excess of hydrochloric acid, dilute, and heat to boiling. While still boiling, add barium chloride solution as long as a precipitate forms and enough barium hydrate to make the liquid strongly alkaline. As soon as the precipitate has settled, filter and wash with hot water, heat the filtrate to boiling, add sufficient ammonium carbonate solution (1 part of ammonium carbonate in 5 parts of 2 per cent ammonia water) to precipitate all the barium, filter, and wash with hot water. Evaporate the filtrate to dryness, ignite below redness to remove ammonia salts. Add to the residue a little water and a few drops of ammonium carbonate solution. Filter into a tared platinum dish, evaporate, ignite below redness, and weigh the mixed potassium and sodium chlorids.

Determine the potash as potassium platinichlorid, using the factors 0.1939 for K_2O and 0.3069 for KCl.

13.—DETERMINATION OF PHOSPHORIC ACID.

Mix 5 grams of the material with a little magnesium-nitrate solution, dry, ignite, and dissolve in hydrochloric acid. In an aliquot of the solution determine phosphoric acid as magnesium pyrophosphate by the molybdic method.^b

14.—DETERMINATION OF SULPHURIC ACID.

Boil 5 grams of the powder gently for one and one-half hours with a mixture of 300 cc of water and 15 cc of concentrated hydrochloric acid. Dilute to 500 cc, draw off an aliquot portion of 100 cc, dilute considerably, precipitate with barium chlorid, filter through a Gooch crucible, ignite, and weigh. Direct solution of the material without burning of the organic matter was proposed by Crampton.^c The dextrose, formed by the action of the acid on the starch of baking powders, does not interfere with the accuracy of the process.^d

15.—DETERMINATION OF AMMONIA.

Ammonia alum is often an ingredient of cream-of-tartar substitutes and baking powders, and ammonium carbonate is occasionally present in baking powders. Determine ammonia by distillation with caustic soda into standard acid and titration.

XVI. FOOD PRESERVATIVES.

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1.—DETECTION OF FORMALDEHYDE.

(a) PREPARATION OF SAMPLE.

If the material be solid or semisolid, macerate from 200 to 300 grams in a mortar with about 100 cc of water until a sufficient degree of fluidity is obtained. Take the sample so prepared and make distinctly acid with phosphoric acid. Transfer to a short-necked distilling flask of copper or glass of from 500 to 800-cc capacity. If a copper flask is used, the heat can be applied directly; if the flask be glass, it is best to heat in a linseed-oil bath. Connect flask with glass condenser and distill off from 40 to 50 cc. In the case of liquids, acidify from 200 to 300 cc with a strong excess of phosphoric acid, and distill as directed under 3.

^aSee Appendix, p. 160.

^bU. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 12.

^cU. S. Dept. of Agr., Div. of Chem., Bul. 13, part 5, p. 596.

^dConn. Agr. Expt. Sta., Rep. 1900, p. 119.

(b) FIRST METHOD OF DETECTION.

To about 5 cc of the distillate described above add 2 or 3 drops of a 1 per cent aqueous solution of phenol; mix and carefully pour it on about the same amount of concentrated sulphuric acid in a test tube, holding the tube so that the solutions will not mix. The presence of one part of formaldehyde in 100,000 parts is indicated by the formation of a crimson color at the plane of union of the solutions. If the formaldehyde be present in greater quantity, a white turbidity, or a light-colored precipitation, will be formed above the coloring.

If organic matter is distilled over, the charring of it by the sulphuric acid may be mistaken for a trace of formaldehyde; but, on allowing the test to stand for twelve hours, the coloration, if due to formaldehyde, will become a whitish turbidity instead of the dark color which appears if due to the charring of organic matter. Some other aldehydes will give the same result, and it is, therefore, not conclusive.

(c) SECOND METHOD OF DETECTION.^a

Add about 5 cc of the distillate obtained under (a) to an equal volume of pure milk in a porcelain casserole, and about 10 cc of concentrated hydrochloric acid containing 1 cc of 10 per cent ferric chlorid solution to each 500 cc of acid. Heat to 80° or 90° directly over the gas flame, holding the casserole by the handle and giving it a rotary motion to break up the curd. A violet coloration indicates formaldehyde.

(d) THIRD METHOD OF DETECTION.

Dissolve 1 gram of phenylhydrazin hydrochlorid and 1.5 grams of sodium acetate in 10 cc of water. To 1 cc of distillate obtained as directed in (d) add 2 drops of reagent and 2 drops of sulphuric acid. If formaldehyde is present, a green color will be produced.

2.—DETECTION OF SULPHUROUS ACID.

Prepare samples as directed under 1 (a), and boil 20 cc of the distillate after the addition of a few drops of bromin or iodine solution. If it is decolorized quickly, test for sulphuric acid with barium chlorid solution.

If sulphurous acid or sulphite is present, determine it quantitatively as directed under wine (page 90).

3.—DETECTION OF SALICYLIC ACID.

If the material be a solid or semisolid, macerate 200 to 300 grams in a mortar with about 400 cc of water made slightly alkaline with sodium or potassium hydroxid, and strain through a cotton bag. Acidify the filtrate with dilute (1:3) sulphuric acid, and extract by shaking with about 30 cc of chloroform or ether.^b Separate from the water with a separatory funnel. If a clear solution is obtained, place the chloroform or ether in a small porcelain dish and evaporate at a low temperature. If an emulsion is formed and a clear solution will not separate out on standing, whirling in a Babcock milk tester or some other centrifugal machine will usually give the desired clear solution. Take up the residue in the porcelain dish with 3 or 4 cc of hot water and divide into two portions, one for salicylic acid and the other for saccharin.

In the case of materials containing large amounts of extractive matter, and those from which the water solution can not be separated from the solid matter by straining, it may be found necessary to separate them by distillation, though straining is always preferable. In such cases acidify the macerated material with phosphoric acid, and transfer to a distilling flask, with a very short neck and wide mouth. An Erlenmeyer flask with inside diameter of mouth $1\frac{1}{4}$ inches is a good shape. The

^a Leach, Twenty-ninth An. Rep. Mass. Board of Health, 1897, p. 558.

^b See appendix, page 160.

tube connecting the flask with condenser should be very short, with an inside diameter of not less than $\frac{3}{8}$ inch, and should turn into the condenser immediately above the stopper in flask. Conduct steam through a small tube passing through the stopper and dipping deeply into the material in the flask. Submerge the distilling flask almost to the stopper in a linseed oil bath and distill with temperature of the oil at from 120° to 130° C.

Care must be taken not to let the contents of the flask get too low, as the heat will decompose the organic matter. The temperature in the flask will go but little above 100° C. unless the solution in the flask is allowed to get too low.

Distill off 500 cc to 600 cc, acidify with dilute sulphuric acid, extract with about 30 cc of chloroform or ether, and proceed as directed above.

(a) SACCHARIN.

If the solution for saccharin and salicylic acid has an intensely sweet taste, it is an indication of saccharin. If it is sweet, dilute a portion of it about ten times, and taste again. (See also page 51.)

(b) SALICYLIC ACID.

(1) *First method.*^a

Place a few drops of solution for salicylic acid in a porcelain dish, add 2 or 3 drops of ferric chlorid solution in such a way that the solutions will come together slowly, which will give a purple or violet color if salicylic acid is present.

(2) *Second method.*^a

Place about 0.5 cc of the solution in a porcelain dish and evaporate to dryness at a low temperature. Warm the residue carefully with one drop of concentrated nitric acid, and add 2 or 3 drops of ammonia until alkaline. The presence of salicylic acid is indicated by the formation of a yellow color of ammonium picrate, and may be confirmed by dyeing a thread of fat-free wool in it.

4.—DETECTION OF BENZOIC ACID.^b

Separate benzoic acid by extraction or distillation as directed under salicylic acid, and test by one of the following methods:

(1) *First method.*^c

Divide solution for benzoic acid into three portions and examine one portion by each of the following methods: Make alkaline with ammonium hydroxid, expel the excess of ammonia by evaporation, take up the residue with water, and add a few drops of a neutral 0.5 per cent solution of ferric chlorid. The presence of benzoic acid will be indicated by the formation of a brownish-colored precipitate of ferric benzoate.

(2) *Second method.*^c

Evaporate to dryness and treat the residue with 2 or 3 cc of strong sulphuric acid, Heat till white fumes appear; organic matter is charred and benzoic acid is converted into sulpho-benzoic acid. A few crystals of potassium nitrate are then added. This causes the formation of metadinitrobenzoic acid. When cool, the acid is diluted with water and ammonia added in excess, followed by a drop or two of ammonium

^a U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 132.

^b See also Appendix, page 160.

^c Bul. Soc. Chim. 1890 [3], 3, 414.

sulphid. The nitrocompound becomes converted into ammonium metadiamidobenzoic acid, which possesses a red color. This reaction takes place immediately, and is seen at the surface of the liquid without stirring.

(3) *Third method.*

Evaporate to dryness at low temperature. If benzoic acid is present in great quantity it will crystallize out in shining leaflets, with characteristic odor. These will melt at 120° C.

5.—DETECTION AND DETERMINATION OF BORIC ACID AND BORATES.

(a) FIRST QUALITATIVE METHOD.^a

Render decidedly alkaline with lime water about 25 grams of the sample and evaporate to dryness on a water bath. Ignite the residue to destroy organic matter. Add about 15 cc of water and hydrochloric acid, drop by drop, to acid reaction. Then add about 1 cc of concentrated hydrochloric acid. Moisten a piece of delicate turmeric paper with the solution; if borax or boric acid is present, the paper on drying will acquire a peculiar red color, which is changed by ammonia to a dark blue-green, but is restored by acid. This color is almost unmistakable, but it is best for one not familiar with it to conduct a blank.

A preliminary test may be made by immersing a strip of turmeric paper in about 100 cc of liquid foods, to which about 7 cc of concentrated hydrochloric acid has been added. Solid and pasty foods may be heated with enough water to make them thoroughly fluid, hydrochloric acid added in about the proportion of 1 to 15, and tested in the same manner.

(b) SECOND QUALITATIVE METHOD.^b

Add an equal volume of fresh saturated turmeric tincture and a drop of hydrochloric acid, and heat a few seconds. If the sample is a liquid, evaporate with the turmeric and heat with a drop of dilute hydrochloric acid for a few seconds; then if borax or boric acid is present a pink or dark red color will appear, depending upon the amount of boric acid present. Cool and add a drop of ammonium hydroxid, when a dark blue-green will appear.

(c) QUANTITATIVE METHOD.^c

Render 100 grams of the sample decidedly alkaline with sodium hydroxid, and evaporate to dryness in a platinum dish. Ignite the residue cautiously, heat with about 20 cc of water, and add hydrochloric acid, drop by drop, until all is dissolved. Transfer to 100-cc flask, the bulk not being allowed to go over 50 cc or 60 cc. Add 0.5 gram of calcium chlorid and a few drops of phenolphthalein, then a 10 per cent solution of caustic soda until a permanent slight pink color is produced, and finally 25 cc of lime water. Make the volume up to 100 cc. Mix well and filter through a dry filter. To 50 cc of the filtrate add normal sulphuric acid till the pink color disappears, then methyl orange, and continue the addition of the acid until the yellow is just changed to pink. Then add fifth-normal caustic soda till the liquid assumes the yellow tinge, excess of soda being avoided. Boil to expel carbon dioxid. Cool the solution, add a little phenolphthalein, and an equal volume of glycerine. Titrate with standardized sodium hydroxid until a permanent pink color is produced.

One cubic centimeter of fifth-normal soda solution is equal to 0.0124 gram crystallized boric acid.

^a U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 134.

^b U. S. Dept. of Agr., Div. of Chem., Bul. 51, p. 113.

^c Thomson's method—Sutton's Volumetric Analysis, page 100.

XVII.—COLORING MATTER.

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1.—GENERAL DISCUSSION.

The food chemist has two problems in connection with coloring matter—the analysis of dyes used for food colors and the detection and identification of the color used in a food. The first will require an estimation of the heavy metals present and a determination of the general group to which the color belongs. The second will require the detection of the presence of the color, the determination of the group to which the color belongs, and the presence or absence of poisonous metals.

The complete examination of dyes is too large a subject to take up in these methods, and one will have to refer to such works as Schultz and Julius, on Organic Coloring, Allen's Commercial Organic Analysis, and others that go into the subject in an exhaustive manner. The determination of the general nature of the dye can be made by the use of Rota's scheme, which is the simplest of the many different methods proposed and is quite satisfactory, although it requires a great deal of care and experience.

The detection of the color in a food product and its identification are more difficult. It must be separated in a somewhat pure condition and then tested. Almost all the methods for separating added color from the food will take up some of the natural color of the food as well.

As will be seen in tables for the extraction of fruit colors (p. 113), amyl alcohol extracts the coloring matter from many fruits, and these extracts may easily be mistaken for added colors.

Some of the highly colored fruit juices will dye wool, and the color will be permanent; but these will not be mistaken for coal-tar dyes if the double-dyeing method is followed.

In the methods of manufacture of coal-tar dyes many become contaminated with poisonous metals, such as arsenic, copper, zinc, tin, and lead. There is always the possibility of the presence of arsenic, as sulphuric acid is used at one stage or another in the preparation of nearly every dye.

Some colors have metallic atoms in their molecule, such as malachite green, which is a double chlorid of zinc in combination with the organic group.

Many vegetable colors are sold as lakes of tin or alum. Other colors are known to have a toxic action, such as picric acid and naphthol yellow.

Mixtures of two or more dyes are often added to foods. This can sometimes be shown by a system of fractional dyeing, where the dyes are taken up at different rates by the fabric. In examining mixtures of red, orange, and blue dyes, which are widely sold for coloring wine, the writer found that the woolen cloth took up the red much faster than the orange, and the blue slowest; so that the first piece of cloth dyed was red; the second, a lighter shade; the third, greenish, and the fourth, bluish.

2.—DETERMINATION OF HEAVY METALS.

Directions for this determination are given under Vegetables (p. 52).

3.—DETERMINATION OF COAL-TAR COLORING MATTERS BY DYEING WOOL.

(a) METHOD OF SOSTEGNI AND CARPENTIERI.^a

From 10 to 20 grams of the sample are dissolved in 100 cc of water, filtered if necessary, acidified with from 2 to 4 cc of 10 per cent solution of hydrochloric acid, and a piece of woolen cloth, which has been washed in a very dilute solution of boiling

^a Ztsch. anal. Chem., 1896, 35, 397; U. S. Dept. of Agr., Div. of Chem., Bul. 46 revised, p. 68.

potassium hydroxid and then washed in water, is immersed in it and boiled for five to ten minutes. The cloth is removed, thoroughly washed in water, and boiled with very dilute hydrochloric acid solution. Then after washing out the acid the color is dissolved in a solution of ammonium hydroxid (1 to 50). With some of the dyes solution takes place quite readily, while with others it is necessary to boil some time. The wool is taken out, a slight excess of hydrochloric acid is added to the solution, another piece of wool is immersed and again boiled. With vegetable coloring matter this second dyeing gives practically no color, and there is no danger of mistaking a fruit color for one of coal-tar origin. It is absolutely necessary that the second dyeing should be made, as some of the coal-tar dyes^a will dye a dirty orange in the first acid bath which might be easily passed for vegetable color, but on solution in alkaline bath the second acid bath dyes a bright pink.

(b) ARATA'S METHOD.^b

This method gives results comparable with those of the first dyeing of the preceding method. It was recommended for detecting coal-tar colors in wine, and has been used by Winton^c in fruit products.

From 20 to 30 grams of the sample dissolved in 100 cc of water are boiled for ten minutes with 10 cc of a 10 per cent solution of potassium bisulphate and a piece of white wool or woollen cloth which has been previously heated to boiling in a very dilute solution of sodium hydroxid and thoroughly washed in water. After removal from the solution the wool is washed in boiling water, and dried between filter papers. If the coloring matters are entirely from the fruit the wool will be either uncolored or will take on a faint pink or brown, which is changed to green or yellow by ammonia and not restored by washing.

In addition to this, it is advisable in all cases to dissolve out the coloring matter with ammonia as in the first method and dye again, since Arata's method gives practically the same results as the first dyeing in hydrochloric acid bath and needs to be substantiated by the second dyeing.

Another advantage in the second dyeing is that if a large piece of woollen cloth is used in the first dyeing, and a small piece in the second dyeing, small amounts of coloring matter can be brought out much more decidedly in the second dyeing where practically all of the vegetable coloring matter has been excluded. The coloring matter can be identified to a certain extent by the schemes of Witt,^d Allen, Weingartner,^e Dommergue,^f Girard^g and Dupre, and Rota.^h The tests can be madeⁱ directly on the dyed fabric or the dye can be dissolved out.^j To remove the color wash the wool with dilute tartaric acid and then with water and dry between filter paper. Saturate the wool with strong sulphuric acid and press out the color with a glass rod after from five to ten minutes and dilute to 10 cc with water.

Remove the wool, make solution alkaline with ammonia, and when cold extract with from 5 to 10 cc of amyl alcohol. Separate the amyl alcohol, evaporate it to dryness, and test the residue with strong sulphuric acid.

Ponceau R, 2R, 3R, S and 3S gives yellow red to carmine red.

Ponceau S and tropaeolin O give yellow to orange yellow.

^a U. S. Dept. of Agr., Bureau of Chem. Bul. 66.

^b Ztschr. anal. Chem., 1889, 28, 639.

^c Conn. Exp. Sta. Report, 1899, Pt. II, p. 131.

^d Ztschr. anal. Chem., 1887, 26, 100.

^e Com. Org. Anal., Vol. III, pt. 1, pp. 399-420.

^f Ztsch. anal. Chem., 1888, 27, 232-249.

^g Ztsch. anal. Chem., 1890, 29, 369-377.

^h Analyse des Matières Alimentaires, etc., 583-593.

ⁱ Analyst, 1899, 24, 41.

^j Ztsch. anal. Chem., 1889, 28, 639; Borgmann, Anal. des Weines, p. 91; Winton, Conn. Expt. Sta. Rept., 1899, Pt. II, p. 131.

Biebrich scarlet gives a green; Bordeaux red and crocein scarlet give blue; tropaeolin 000 and solid red give violet.

If the wool is well dyed most of these colors may be obtained on the fabric.

This gives only the reactions of a few of the more common colors. In order to carry the work farther the more complete works referred to will have to be used.

4.—DETECTION OF COAL-TAR COLORS BY EXTRACTION WITH SOLVENTS.

In the Paris Municipal Laboratory^a the following scheme of extraction of coal-tar colors is used:

The acid colors, sulphu-fuchsin, azo derivatives, and phthaleins are not precipitated by tannin and are insoluble or only slightly soluble in acetic ether or amyl alcohol.

The basic colors (fuchsin, safranin, etc.) are precipitated by tannin and readily soluble in acetic ether or amyl alcohol.

I. To 50 cc of wine add ammonium hydroxid in slight excess; then add 15 cc of amyl alcohol, shake, and allow to stand.

1. If the alcohol be colored red or violet, decant, wash, filter, evaporate to dryness in presence of a piece of wool, and test the dyed wool with sulphuric acid.

2. If the alcohol be not colored, separate, and add acetic acid. If the alcohol becomes colored the presence of basic aniline color is indicated.

3. If the amyl alcohol is uncolored, both before and after the addition of acetic acid, no basic coal-tar color is present.

II. Add an excess of calcined magnesia and then a 20 per cent solution of mercuric acetate and bring to a boil. A coloration before or after addition of acetic acid indicates the presence of coal-tar dyes, particularly acid dyes.

III. Extract the solution with acetic ether made alkaline by barium hydroxid. This dissolves basic colors.

In any case the colors must be fixed on wool, as many of the fruit colors are extracted and will give reactions with sulphuric acid, which may be mistaken for coal-tar colors.

The extraction of fruit colors is shown in the following tables, the first of which was prepared by Truchon and Martin-Claude,^b and the second by the writer. The fresh fruit juice was very slightly acidified by hydrochloric acid before extraction. In no case in the dyeing test was there any danger of mistaking the vegetable color for one of coal-tar origin where the double-dyeing method was used.

Extraction of fruit colors with amyl-alcohol.

Fruit.	Coloration of acid solution. ^c		Coloration of ammoniacal solution.		Addition of a drop of H ₂ SO ₄ to dyed fabric.
	Juice.	Amyl-alcohol extract.	Juice.	Amyl-alcohol extract.	
Early cherries	Red	Yellow	Green	Uncolored	Yellow.
Ripe cherries	Red	Uncolored ..	Green	Uncolored	Yellow.
Early strawberries	Red	Rose	Green	Uncolored	Rose.
Ripe strawberries	Red	Red	Green	Uncolored	Rose (dyes silk a rose red).
Raspberries	Red	Red	Green	Uncolored	Dyes silk rose. Uncolored.
Red currants	Red	Uncolored ..	Green	Uncolored	
White currants	White	Uncolored ..	Brown	Uncolored	
Black currants	Dark red ..	Red	Deep green ..	Uncolored	
Peaches	Yellow	Uncolored ..	Brown	Yellow-red	
Pears	Yellow	Uncolored ..	Brown	Yellow-red	Dyes silk rose. Uncolored.
Quinces	Yellow	Uncolored ..	Brown	Yellow-red	
Apples	Yellow	Uncolored ..	Brown	Yellow-red	
Apricots	Yellow	Uncolored ..	Brown	Yellow-red	
Green gage plums	Yellow	Uncolored ..	Brown	Yellow-red	

^a Girard and Dupré *Analyse des Matières*, etc., p. 167

^b *Journ. pharm. chim.*, 1901, 13, 174.

^c Acidity of the juice.

Extraction of fruit colors with amyl-alcohol and with ether.

Fruit.	Color with NH_4OH .	Ether extract from acid solution.	Amyl-alcohol extract from acid solution.	Dyeing tests on the juice.
Strawberry	Purple.....	None	Deep red	Color washed out.
Red raspberry	Purple.....	None	Deep red	All color does not wash out, but does not dye in the second acid bath.
Blackberry	Blue-purple ..	None	Very deep red.	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Cherry.....	Purple.....	None	Red	Do.
Blackberry	Blue-purple ..	None	Red	Do.
Wild dewberry	Blue-purple ..	None	Red	Do.
Currant.....	Blue-purple ..	None	Red	Do.

It will be seen from these two tables that amyl-alcohol, as a rule, extracts fruit coloring matter from acid solution, while ether does not. Neither amyl-alcohol nor ether extracted any color from alkaline solution of the fruit juices.

5. DETERMINATION OF ACID MAGENTA—GIRARD'S METHOD.^a

Add to 100 cc of the solution to be tested 2 cc of potassium hydroxid (5 to 100). If this does not neutralize the acid, add enough to do it. Then add 4 cc of mercuric acetate (10 to 100), agitate and filter. The filtrate should be colorless and slightly alkaline. Acidify with a slight excess of dilute sulphuric acid, and if the solution remains uncolored there is no acid magenta present. If it becomes a light violet-red and there has been no other dye shown by the amyl-alcohol extracts, the presence of acid magenta is shown.

Acid magenta in acid solution dyes wool a magenta red. Wool dyed with it is turned yellow by strong hydrochloric acid, decolorized by ammonium hydroxid, and regains its color when washed with water.

6.—TEST FOR MARTIUS YELLOW OR NAPHTHALENE YELLOW.

Extract with 95 per cent alcohol from an acidulated sample. Evaporate the alcoholic solution to dryness with a piece of wool, which will be dyed a bright yellow, and test the dyed wool. Both sulphuric and hydrochloric acids completely decolorize it.

7.—ROTA'S METHOD OF IDENTIFICATION OF ORGANIC COLORING MATTER.^b

The coloring matters are divided into four groups by the use of stannous chlorid and hydrochloric acid and of caustic potash.

The reagents are a 10 per cent solution of stannous chlorid and a 20 per cent solution of caustic potash.

Dilute the aqueous or alcoholic solution of coloring matter to about 1 to 10,000. This strength is not of vital importance, but the color must not be too deep, as it will mask the reduction in some cases, such as the safranins, where it is slow and not complete. Add to the solution a few drops of stannous chlorid and a few drops of hydrochloric acid; shake, and heat to boiling. Care must be taken to carry along for comparison a solution of the coloring matter acidified with hydrochloric acid, in order not to mistake the action of the acid alone for reduction. Some of the colors—for instance, safranins and indulins—are slow to be reduced and must be allowed to stand for some time. For the stannous chlorid and hydrochloric acid can be substituted a solution of tin in strong hydrochloric acid.

^a Girard & Dupré, *Analyse des Matières, Alimentaires, etc.*, p. 169; Winton, Conn. Expt. Sta. Rept., 1899, Pt. II, 132.

^b Chem. Ztg., 1898, 22, 437-442; Analyst, 1899, 24, 41.

As soon as the group is determined it is possible to carry the work further by reference to tables of coloring matter^a in which the physical, chemical, and tinctorial properties are given; but it is impossible for the published books to keep up with the new dyes which are constantly being discovered, so that the tables are never complete, although they will, as a rule, contain all the data necessary.

Classification of organic coloring matters.

[A portion of the aqueous or alcoholic solution is treated with HCl and SnCl₂.]

Complete decolorization. Reducible coloring matters. Colorless solution is treated with Fe ₂ Cl ₆ or shaken, with exposure to air.		The color changed no further than with HCl alone. Nonreducible colors. A part of original solution is mixed with 20 per cent KOH and warmed.	
The liquid remains unchanged. Not reoxidizable coloring matters.	The original color restored. Reoxidizable coloring matters.	Decolorization or a precipitate. Imido-carboquinone coloring matters.	No precipitation. Liquid becomes more colored. Oxy-carboquinone coloring matters.
CLASS I.	CLASS II.	CLASS III.	CLASS IV.
Nitro, nitroso, and azo colors, including azoxy and hydrazo colors. Pieric acid, naphthol yellow, Ponceau, Bordeaux, and Congo red.	Indogenide and imidoquinone coloring matters, methylene blue, safranin, indigo-carmin.	Amido-derivatives of di and triphenylmethane, auramines, acridines, quinolines, and color derivatives of thio benzenil. Fuchsin, rosaniline, auramine.	Nonamide diphenylmethane, oxy-ketone, and most of natural organic coloring matters. Eosines, aurin, alizarin.

^aSchultz and Julius, Tabellarische Übersicht der künstlichen organischen Farbstoffe; Allen, Commercial Organic Analysis, 3d ed., Vol. III, pt. 1, pp. 529-565.

Characteristics of organic coloring matters.

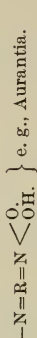
CLASS I.—REDUCED BY $\text{HCl} + \text{SnCl}_2$ AND NOT REOXIDIZABLE.

Nitro-coloring matters.



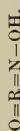
Yellow or orange, soluble in water. Wool and silk dyed directly, but not cotton. The aqueous solution shows tendency to decolorization with HCl . With $\text{HCl} + \text{SnCl}_2$ partially reduced, giving red nitro-amido derivatives (nitramines) or nitro-phenols turning red in KOH .

Nitramines; soluble in ether in the presence of KOH .



Nitro-phenols; insoluble in ether in the presence of acetic acid. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Nonsulphonated; soluble in ether in presence of acetic acid. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Victoria yellow. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Sulphonated; insoluble in ether. Naphthol yellow.

Nitro-coloring matters.



Brown or green, usually insoluble in water; indirect for fibres; with $\text{H}_2\text{SO}_4 + \text{CHOH}$ give blue color (Liebermann's reaction).

Nonsulphonated; insoluble in water; soluble in alcohol; soluble in ether in presence of acetic acid. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Dioxine (L).

Sulphonated; soluble in water; insoluble in ether. Naphthol green.

Azo-coloring matters.



Their aqueous solution decomposed with KOH and extracted with ether gives an ethereal extract with annexed characteristics.

Colored; shaken with dilute acetic acid yields to it the original color. Basic coloring matters. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Nonsulphonated amido-azo coloring matters. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Oxyazo coloring matter without carbonyl.

Colored solution; not yielding its color to dilute acetic acid. Neutral coloring matters. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Oxyazo coloring matters with carbonyl group.

Colorless solution; yields nothing to acetic acid. Acid coloring matter. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Nonsulphonated; extracted by ether from dilute solution in acetic acid. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Oxyazo coloring matters with carbonyl group.

Sulphonated; not extracted by ether from solution in dilute acetic acid. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Nonamido compounds unaltered by HNO_3 . $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Amido compounds changed by HNO_3 .

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Sudan 1 (A).

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Diamond yellow (By).

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Chrysamine.

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Bordeaux B (A).

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Azo blue (A).

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Solid yellow N (P).

Indirect for cotton wool. Direct for cotton wool. $\left. \vphantom{\begin{smallmatrix} \text{O} \\ \diagup \end{smallmatrix}} \right\}$ Congo red (A).

Characteristics of organic coloring matters—Continued.

 CLASS II.—REDUCED BY $\text{HCl} + \text{SnCl}_2$ AND REOXIDIZABLE.

The ethereal solution is colored or colorless, and yields the original color to 5 per cent acetic acid. Basic coloring matters fixed on wool in alkaline bath.	The solution is readily reduced by $\text{HCl} + \text{SnCl}_2$ in the cold.	Oxyazines (no sulphur).	Nile blue A (B).
Colored; does not yield the color to acetic acid. Neutral coloring matters. Insoluble in water. Soluble in alcohol. Fixed on fibers in bath.	The colored solution is reduced but slowly and incompletely even on warming, and with the addition of much $\text{SnCl}_2 + \text{HCl}$.	Thiazines (sulphur).	Methylene blue.
Uncolored; yields nothing to acetic acid. Acid coloring matters. Soluble in water. Fixed on wool in acid bath.	Blue coloring matters changed by HCl on warming. Red or blue coloring matters. Unaltered by HCl. With HNO_3 yield isatin.	Indulines; blue color with conc. H_2SO_4. Blue on dilution. Safranins; green color with H_2SO_4. On dilution blue; then violet.	Induline soluble in alcohol. Safranin T, extra (A).
The ethereal solution washed with water has the annexed characteristics.	Indophenols.	Indogenides.	Indophenol.
The aqueous or alcoholic solution is treated with KOH and extracted with ether.	Oxazones.	Reduced by $\text{SnCl}_2 + \text{HCl}$. Not reduced by $\text{SnCl}_2 + \text{HCl}$.	Indigotin.
			Fluorescent blue; orefer.
			Sulphonated indogenides. Sulphonated thiazines. Sulphonated indulines.
			Indigo carmine. Thiocarmine R (C). Soluble nigrosin.

Characteristics of organic coloring matters—Continued.

CLASS IV.—COLORING MATTERS NOT REDUCED BY $\text{SnCl}_2 + \text{HCl}$. CONTAINING THE OXY-QUINONE CARBON CHROMOPHORE $\text{O}=\text{R}=\text{O}=\text{}$.

The alcoholic solution of the coloring matter treated with a few drops of a dilute solution of FeCl_3 (1:1000).	Remains unaltered. Nonamido, triphenyl- methane coloring matters. Usually soluble in water and direct for wool.	The coloring matter is dissolved or suspended in boiling water.	Not directly fixed on wool. Most of them in- soluble in water. Soluble in alcohol without fluorescence.	Aurins.	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{C}=\text{R}^1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$	Aurin.
	Changes to green or olive green.	The original coloring matter is treated with faintly alkaline water is (KOH, 1 per cent).	Dissolves with yellow or reddish yellow color. Monoketones.	Fixed directly on wool. Most of them soluble in water and alcohol. Fluorescent.	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{C}=\text{R}^1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$	Eosin.
Oxy-ketone coloring matters. Most of them insoluble in water and indirect for fibers.	Dissolves with red, reddish violet, green, or blue color. Diketones (quinones).	The alkaline so- lution treated with excess of HCl.	Inclined to decolorization, especially on warming (with decomposition).	Benzophenones.	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{R} \end{array}$	Alizarin yellow A (B).
			Colored intense yellow with- out decomposition.	Flavones.	$\begin{array}{c} \text{O} \\ \diagup \\ \text{R} \\ \diagdown \\ \text{CO} \end{array}$	Quercetin.
		The alkaline solution acid- ified with acetic acid.	The free coloring matter pre- cipitated. Usually soluble in ether and indirect for fibers.	Nonsulphonated anthraquinones.	$\begin{array}{c} \text{O} \\ \diagup \\ \text{R} \\ \diagdown \\ \text{CO} \end{array}$	Alizarin.
			Coloring matters remain in solution. Insoluble in ether fixed directly on wool.	Sulphonated an- thraquinones.	$\begin{array}{c} \text{CO} \\ \diagup \\ \text{R} \\ \diagdown \\ \text{CO} \end{array}$	Sulphonated al- izarin (alizarin red).

8.—DETERMINATION OF VEGETABLE COLORS.

A great many tests for vegetable colors are given, depending largely on color reactions with different reagents, but these must be used with very great discrimination, as they depend very largely on a fine judgment of shades of colors which many eyes are not able to distinguish.

A great deal of work has been done on detection of vegetable colors,^a but only in a very few cases are the reactions specific enough to be decisive.

9.—DETECTION OF TURMERIC.^b

Extract the color with alcohol. Dip a piece of filter paper into this tincture and dry at 100° C.

Then moisten in a weak solution of boric acid to which a few drops of hydrochloric acid have been added. On drying this, a cherry red color will be developed in the presence of turmeric which is characteristic.

10.—DETECTION OF CARAMEL.

AMTHOR TEST.^c

Ten cubic centimeters of the solution to be tested are put into a high, narrow glass with perpendicular sides, as, for example, a small bottle; add from 30 to 50 cc of paraldehyde, depending on the intensity of the coloring, and enough absolute alcohol to make the solutions mix. In the presence of caramel a brownish yellow to dark-brown precipitate will collect in the bottom of the glass. Decant the liquor, wash once with absolute alcohol, dissolve in small amount of hot water, and filter. The color of this will give some idea as to the amount of caramel present.

It is not allowable to concentrate a solution by evaporation on a steam bath, as caramel may be formed; if it is necessary to concentrate it must be done over sulphuric acid or at diminished pressure.

In order to further identify the color it is poured into a freshly prepared solution of phenylhydrazin (2 parts phenylhydrazin-hydrochlorid, 3 parts sodium acetate, and 20 parts of water). The presence of a considerable quantity of caramel gives a dark-brown precipitate in the cold, which is hastened by heating a little.

In the case of a very small amount it takes some hours for it to collect.

11.—DETECTION OF COCHINEAL.

Cochineal is used to a certain extent as a coloring matter in foods, and a very satisfactory test for it is that given in Girard and Dupré.^d Dissolve the food product in water, filtering if necessary. Acidulate with hydrochloric acid and extract with amyl alcohol, which becomes colored more or less yellow or orange, depending on the quantity of cochineal present. Separate the amyl alcohol and wash until neutral. Then separate into two portions; to the first add drop by drop a very dilute solution of uranium acetate, shaking thoroughly after each addition. In the presence of cochineal a characteristic emerald-green color is produced.^e

To the second portion add a drop or so of ammonia, and in presence of cochineal a violet coloration results. This, however, is not so sensitive to very small amounts as the first tests, and many fruit colors give tests hardly to be distinguished.

Cochineal carmine is liable to contain tin, as it is often a tin lake, although alum is also used. It is also liable to adulteration with lead compounds.

^a Girard and Dupré, *Analyse des Matières Alimentaires*, etc., 580-581, also 169; A. W. Blythe, *Foods, their Comp. and Anal.*, p. 91-109; Allen *Com. Org. Anal.*, Vol. III, Pt. I; E. Brueher, *Fals. Subst. Alim.*, p. 162; W. Lenz, *Ztschr. anal. Chem.*, 1885, **24**, 285.

^b Allen, *Com. Org. Anal.*, Vol. III, Pt. I, p. 359; U. S. Dept. of Agr., Div. of Chem., *Bul.* 51, p. 131.

^c Ztseh. *anal. Chem.*, 1885, **24**, 30; Borgemann, *Anal. des Weines*, p. 98.

^d *Analyse des Matières Alimentaires*, etc., p. 580.

^e The writer has tested this reaction on a number of amyl alcohol extracts from fruits, and in no case was there any chance of mistake in the reaction. Most fruits give a brown color, while blackberries and currants give a bluish color.

REFERENCE TABLES.

TABLE I.—*Specific gravity and percentage of alcohol.*

[According to Squibb.]

Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.		Per cent alcohol by volume.	Specific gravity.	
	At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.		At 15.56° C.	At 25° C.
1	0.9985	0.9970	36	0.9578	0.9521	71	0.8875	0.8796
2	.9970	.9953	37	.9565	.9507	72	.8850	.8771
3	.9956	.9938	38	.9550	.9489	73	.8825	.8746
4	.9942	.9922	39	.9535	.9473	74	.8799	.8719
5	.9930	.9909	40	.9519	.9456	75	.8769	.8689
6	.9914	.9893	41	.9503	.9438	76	.8745	.8665
7	.9898	.9876	42	.9490	.9424	77	.8721	.8641
8	.9890	.9868	43	.9470	.9402	78	.8696	.8616
9	.9878	.9855	44	.9452	.9382	79	.8664	.8583
10	.9869	.9846	45	.9434	.9363	80	.8639	.8558
11	.9855	.9831	46	.9416	.9343	81	.8611	.8530
12	.9841	.9816	47	.9396	.9323	82	.8581	.8500
13	.9828	.9801	48	.9381	.9307	83	.8557	.8476
14	.9821	.9793	49	.9362	.9288	84	.8526	.8444
15	.9815	.9787	50	.9343	.9267	85	.8496	.8414
16	.9802	.9773	51	.9323	.9246	86	.8466	.8384
17	.9789	.9759	52	.9303	.9226	87	.8434	.8352
18	.9778	.9746	53	.9283	.9205	88	.8408	.8326
19	.9766	.9733	54	.9262	.9184	89	.8373	.8291
20	.9760	.9726	55	.9242	.9164	90	.8340	.8258
21	.9753	.9719	56	.9221	.9143	91	.8305	.8223
22	.9741	.9706	57	.9200	.9122	92	.8272	.8191
23	.9728	.9692	58	.9178	.9100	93	.8237	.8156
24	.9716	.9678	59	.9160	.9081	94	.8199	.8118
25	.9709	.9668	60	.9135	.9056	95	.8164	.8083
26	.9698	.9655	61	.9113	.9034	96	.8125	.8044
27	.9691	.9646	62	.9090	.9011	97	.8084	.8003
28	.9678	.9631	63	.9069	.8989	98	.8041	.7960
29	.9665	.9617	64	.9047	.8969	99	.7995	.7914
30	.9652	.9603	65	.9025	.8947	100	.7946	.7865
31	.9643	.9594	66	.9001	.8923			
32	.9631	.9582	67	.8973	.8895			
33	.9618	.9567	68	.8949	.8870			
34	.9609	.9556	69	.8925	.8846			
35	.9593	.9538	70	.8900	.8821			

TABLE II.—*Percentage of alcohol.*

[Recalculated from the determinations of Gilpin, Drinkwater, and Squibb.]

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
1.00000	0.00	0.00	0.00	0.99884	0.75	0.60	0.60	0.99775	1.50	1.19	1.19
0.99992	0.05	0.04	0.04	.99877	0.80	0.64	0.64	.99768	1.55	1.23	1.23
.99984	0.10	0.08	0.08	.99869	0.85	0.67	0.67	.99760	1.60	1.27	1.27
.99976	0.15	0.12	0.12	.99861	0.90	0.71	0.71	.99753	1.65	1.31	1.31
.99968	0.20	0.16	0.16	.99854	0.95	0.75	0.75	.99745	1.70	1.35	1.35
.99961	0.25	0.20	0.20	.99849	1.00	0.79	0.79	.99738	1.75	1.39	1.39
.99953	0.30	0.24	0.24	.99842	1.05	0.83	0.83	.99731	1.80	1.43	1.43
.99945	0.35	0.28	0.28	.99834	1.10	0.87	0.87	.99723	1.85	1.47	1.47
.99937	0.40	0.32	0.32	.99827	1.15	0.91	0.91	.99716	1.90	1.51	1.51
.99930	0.45	0.36	0.36	.99819	1.20	0.95	0.95	.99708	1.95	1.55	1.55
.99923	0.50	0.40	0.40	.99812	1.25	0.99	0.99	.99701	2.00	1.59	1.59
.99915	0.55	0.44	0.44	.99805	1.30	1.03	1.03	.99694	2.05	1.63	1.62
.99907	0.60	0.48	0.48	.99797	1.35	1.07	1.07	.99687	2.10	1.67	1.66
.99900	0.65	0.52	0.52	.99790	1.40	1.11	1.11	.99679	2.15	1.71	1.70
.99892	0.70	0.56	0.56	.99782	1.45	1.15	1.15	.99672	2.20	1.75	1.74

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.
0.99665	2.25	1.79	1.78	0.99215	5.50	4.40	4.37	0.98807	8.75	7.03	6.95
.99658	2.30	1.83	1.82	.99208	5.55	4.44	4.40	.98801	8.80	7.07	6.99
.99651	2.35	1.87	1.86	.99202	5.60	4.48	4.44	.98795	8.85	7.11	7.03
.99643	2.40	1.91	1.90	.99195	5.65	4.52	4.48	.98789	8.90	7.15	7.07
.99636	2.45	1.95	1.94	.99189	5.70	4.56	4.52	.98783	8.95	7.19	7.11
.99629	2.50	1.99	1.98	.99182	5.75	4.60	4.56	.98777	9.00	7.23	7.14
.99622	2.55	2.03	2.02	.99175	5.80	4.64	4.60	.98771	9.05	7.27	7.18
.99615	2.60	2.07	2.06	.99169	5.85	4.68	4.64	.98765	9.10	7.31	7.22
.99607	2.65	2.11	2.10	.99162	5.90	4.72	4.68	.98759	9.15	7.35	7.26
.99600	2.70	2.15	2.14	.99156	5.95	4.76	4.72	.98754	9.20	7.39	7.30
.99593	2.75	2.19	2.18	.99149	6.00	4.80	4.76	.98748	9.25	7.43	7.34
.99586	2.80	2.23	2.22	.99143	6.05	4.84	4.80	.98742	9.30	7.48	7.38
.99579	2.85	2.27	2.26	.99136	6.10	4.88	4.84	.98736	9.35	7.52	7.42
.99571	2.90	2.31	2.30	.99130	6.15	4.92	4.88	.98730	9.40	7.56	7.46
.99564	2.95	2.35	2.34	.99123	6.20	4.96	4.92	.98724	9.45	7.60	7.50
.99557	3.00	2.39	2.38	.99117	6.25	5.00	4.96	.98719	9.50	7.64	7.54
.99550	3.05	2.43	2.42	.99111	6.30	5.05	5.00	.98713	9.55	7.68	7.58
.99543	3.10	2.47	2.46	.99104	6.35	5.09	5.04	.98707	9.60	7.72	7.62
.99536	3.15	2.51	2.50	.99098	6.40	5.13	5.08	.98701	9.65	7.76	7.66
.99529	3.20	2.55	2.54	.99091	6.45	5.17	5.12	.98695	9.70	7.80	7.70
.99522	3.25	2.59	2.58	.99085	6.50	5.21	5.16	.98689	9.75	7.84	7.74
.99515	3.30	2.64	2.62	.99079	6.55	5.25	5.20	.98683	9.80	7.88	7.78
.99508	3.35	2.68	2.66	.99072	6.60	5.29	5.24	.98678	9.85	7.92	7.82
.99501	3.40	2.72	2.70	.99066	6.65	5.33	5.28	.98672	9.90	7.96	7.85
.99494	3.45	2.76	2.74	.99059	6.70	5.37	5.32	.98666	9.95	8.00	7.89
.99487	3.50	2.80	2.78	.99053	6.75	5.41	5.36	.98660	10.00	8.04	7.93
.99480	3.55	2.84	2.82	.99047	6.80	5.45	5.40	.98654	10.05	8.08	7.97
.99473	3.60	2.88	2.86	.99040	6.85	5.49	5.44	.98649	10.10	8.12	8.01
.99466	3.65	2.92	2.90	.99034	6.90	5.53	5.48	.98643	10.15	8.16	8.05
.99459	3.70	2.96	2.94	.99027	6.95	5.57	5.52	.98637	10.20	8.20	8.09
.99452	3.75	3.00	2.98	.99021	7.00	5.61	5.56	.98632	10.25	8.24	8.13
.99445	3.80	3.04	3.02	.99015	7.05	5.65	5.60	.98626	10.30	8.29	8.17
.99438	3.85	3.08	3.06	.99009	7.10	5.69	5.64	.98620	10.35	8.33	8.21
.99431	3.90	3.12	3.10	.99002	7.15	5.73	5.68	.98614	10.40	8.37	8.25
.99424	3.95	3.16	3.14	.98996	7.20	5.77	5.72	.98609	10.45	8.41	8.29
.99417	4.00	3.20	3.18	.98990	7.25	5.81	5.76	.98603	10.50	8.45	8.33
.99410	4.05	3.24	3.22	.98984	7.30	5.86	5.80	.98597	10.55	8.49	8.37
.99403	4.10	3.28	3.26	.98978	7.35	5.90	5.84	.98592	10.60	8.53	8.41
.99397	4.15	3.32	3.30	.98971	7.40	5.94	5.88	.98586	10.65	8.57	8.45
.99390	4.20	3.36	3.34	.98965	7.45	5.98	5.92	.98580	10.70	8.61	8.49
.99383	4.25	3.40	3.38	.98959	7.50	6.02	5.96	.98575	10.75	8.65	8.53
.99376	4.30	3.44	3.42	.98953	7.55	6.06	6.00	.98569	10.80	8.70	8.57
.99369	4.35	3.48	3.46	.98947	7.60	6.10	6.04	.98563	10.85	8.74	8.61
.99363	4.40	3.52	3.50	.98940	7.65	6.14	6.07	.98557	10.90	8.78	8.65
.99356	4.45	3.56	3.54	.98934	7.70	6.18	6.11	.98552	10.95	8.82	8.69
.99349	4.50	3.60	3.58	.98928	7.75	6.22	6.15	.98546	11.00	8.86	8.73
.99342	4.55	3.64	3.62	.98922	7.80	6.26	6.19	.98540	11.05	8.90	8.77
.99335	4.60	3.68	3.66	.98916	7.85	6.30	6.23	.98535	11.10	8.94	8.81
.99329	4.65	3.72	3.70	.98910	7.90	6.34	6.27	.98529	11.15	8.98	8.85
.99322	4.70	3.76	3.74	.98903	7.95	6.38	6.31	.98524	11.20	9.02	8.89
.99315	4.75	3.80	3.77	.98897	8.00	6.42	6.35	.98518	11.25	9.07	8.93
.99308	4.80	3.84	3.81	.98891	8.05	6.46	6.39	.98513	11.30	9.11	8.97
.99301	4.85	3.88	3.85	.98885	8.10	6.50	6.43	.98507	11.35	9.15	9.01
.99295	4.90	3.92	3.89	.98879	8.15	6.54	6.47	.98502	11.40	9.19	9.05
.99288	4.95	3.96	3.93	.98873	8.20	6.58	6.51	.98496	11.45	9.23	9.09
.99281	5.00	4.00	3.97	.98867	8.25	6.62	6.55	.98491	11.50	9.27	9.13
.99274	5.05	4.04	4.01	.98861	8.30	6.67	6.59	.98485	11.55	9.31	9.17
.99268	5.10	4.08	4.05	.98855	8.35	6.71	6.63	.98479	11.60	9.35	9.21
.99261	5.15	4.12	4.09	.98849	8.40	6.75	6.67	.98474	11.65	9.39	9.25
.99255	5.20	4.16	4.13	.98843	8.45	6.79	6.71	.98468	11.70	9.43	9.29
.99248	5.25	4.20	4.17	.98837	8.50	6.83	6.75	.98463	11.75	9.47	9.32
.99241	5.30	4.24	4.21	.98831	8.55	6.87	6.79	.98457	11.80	9.51	9.36
.99235	5.35	4.28	4.25	.98825	8.60	6.91	6.83	.98452	11.85	9.55	9.40
.99228	5.40	4.32	4.29	.98819	8.65	6.95	6.87	.98446	11.90	9.59	9.44
.99222	5.45	4.36	4.33	.98813	8.70	6.99	6.91	.98441	11.95	9.63	9.48

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.98435	12.00	9.67	9.52	0.98088	15.25	12.33	12.10	0.97758	18.50	15.02	14.68
.98430	12.05	9.71	9.56	.98083	15.30	12.38	12.14	.97753	18.55	15.06	14.72
.98424	12.10	9.75	9.60	.98078	15.35	12.42	12.18	.97748	18.60	15.10	14.76
.98419	12.15	9.79	9.64	.98073	15.40	12.46	12.22	.97743	18.65	15.14	14.80
.98413	12.20	9.83	9.68	.98068	15.45	12.50	12.26	.97738	18.70	15.18	14.84
.98408	12.25	9.87	9.72	.98063	15.50	12.54	12.30	.97733	18.75	15.22	14.88
.98402	12.30	9.92	9.76	.98057	15.55	12.58	12.34	.97728	18.80	15.27	14.92
.98397	12.35	9.96	9.80	.98052	15.60	12.62	12.37	.97723	18.85	15.31	14.96
.98391	12.40	10.00	9.84	.98047	15.65	12.66	12.41	.97718	18.90	15.35	15.00
.98386	12.45	10.04	9.88	.98042	15.70	12.70	12.45	.97713	18.95	15.39	15.04
.98381	12.50	10.08	9.92	.98037	15.75	12.75	12.49	.97708	19.00	15.43	15.08
.98375	12.55	10.12	9.96	.98032	15.80	12.79	12.53	.97703	19.05	15.47	15.11
.98370	12.60	10.16	10.00	.98026	15.85	12.83	12.57	.97698	19.10	15.51	15.15
.98364	12.65	10.20	10.03	.98021	15.90	12.87	12.61	.97693	19.15	15.55	15.19
.98359	12.70	10.24	10.07	.98016	15.95	12.91	12.65	.97688	19.20	15.59	15.23
.98353	12.75	10.28	10.11	.98011	16.00	12.95	12.69	.97683	19.25	15.63	15.27
.98348	12.80	10.33	10.15	.98005	16.05	12.99	12.73	.97678	19.30	15.68	15.31
.98342	12.85	10.37	10.19	.98001	16.10	13.03	12.77	.97673	19.35	15.72	15.35
.98337	12.90	10.41	10.23	.97996	16.15	13.08	12.81	.97668	19.40	15.76	15.39
.98331	12.95	10.45	10.27	.97991	16.20	13.12	12.85	.97663	19.45	15.80	15.43
.98326	13.00	10.49	10.31	.97986	16.25	13.16	12.89	.97658	19.50	15.84	15.47
.98321	13.05	10.53	10.35	.97980	16.30	13.20	12.93	.97653	19.55	15.88	15.51
.98315	13.10	10.57	10.39	.97975	16.35	13.24	12.97	.97648	19.60	15.93	15.55
.98310	13.15	10.61	10.43	.97970	16.40	13.29	13.01	.97643	19.65	15.97	15.59
.98305	13.20	10.65	10.47	.97965	16.45	13.33	13.05	.97638	19.70	16.01	15.63
.98299	13.25	10.69	10.51	.97960	16.50	13.37	13.09	.97633	19.75	16.05	15.67
.98294	13.30	10.74	10.55	.97955	16.55	13.41	13.13	.97628	19.80	16.09	15.71
.98289	13.35	10.78	10.59	.97950	16.60	13.45	13.17	.97623	19.85	16.14	15.75
.98283	13.40	10.82	10.63	.97945	16.65	13.49	13.21	.97618	19.90	16.18	15.79
.98278	13.45	10.86	10.67	.97940	16.70	13.53	13.25	.97613	19.95	16.22	15.83
.98273	13.50	10.90	10.71	.97935	16.75	13.57	13.29	.97608	20.00	16.26	15.87
.98267	13.55	10.94	10.75	.97929	16.80	13.62	13.33	.97603	20.05	16.30	15.91
.98262	13.60	10.98	10.79	.97924	16.85	13.66	13.37	.97598	20.10	16.34	15.95
.98256	13.65	11.02	10.83	.97919	16.90	13.70	13.41	.97593	20.15	16.38	15.99
.98251	13.70	11.06	10.87	.97914	16.95	13.74	13.45	.97588	20.20	16.42	16.03
.98246	13.75	11.11	10.91	.97909	17.00	13.78	13.49	.97583	20.25	16.46	16.06
.98240	13.80	11.15	10.95	.97904	17.05	13.82	13.53	.97578	20.30	16.51	16.10
.98235	13.85	11.19	10.99	.97899	17.10	13.86	13.57	.97573	20.35	16.56	16.14
.98230	13.90	11.23	11.03	.97894	17.15	13.90	13.61	.97568	20.40	16.59	16.18
.98224	13.95	11.27	11.07	.97889	17.20	13.94	13.65	.97563	20.45	16.63	16.22
.98219	14.00	11.31	11.11	.97884	17.25	13.98	13.69	.97558	20.50	16.67	16.26
.98214	14.05	11.35	11.15	.97879	17.30	14.03	13.73	.97553	20.55	16.71	16.30
.98209	14.10	11.39	11.19	.97874	17.35	14.07	13.77	.97547	20.60	16.75	16.34
.98203	14.15	11.43	11.23	.97869	17.40	14.11	13.81	.97542	20.65	16.80	16.38
.98198	14.20	11.47	11.27	.97864	17.45	14.15	13.85	.97537	20.70	16.84	16.42
.98193	14.25	11.52	11.31	.97859	17.50	14.19	13.89	.97532	20.75	16.88	16.46
.98188	14.30	11.56	11.35	.97853	17.55	14.23	13.92	.97527	20.80	16.92	16.50
.98182	14.35	11.60	11.39	.97848	17.60	14.27	13.96	.97522	20.85	16.96	16.54
.98177	14.40	11.64	11.43	.97843	17.65	14.31	14.00	.97517	20.90	17.01	16.58
.98172	14.45	11.68	11.47	.97838	17.70	14.35	14.04	.97512	20.95	17.05	16.62
.98167	14.50	11.72	11.51	.97833	17.75	14.40	14.08	.97507	21.00	17.09	16.66
.98161	14.55	11.76	11.55	.97828	17.80	14.44	14.12	.97502	21.05	17.13	16.70
.98156	14.60	11.80	11.59	.97823	17.85	14.48	14.16	.97497	21.10	17.17	16.74
.98151	14.65	11.84	11.63	.97818	17.90	14.52	14.20	.97492	21.15	17.22	16.78
.98146	14.70	11.88	11.67	.97813	17.95	14.56	14.24	.97487	21.20	17.26	16.82
.98140	14.75	11.93	11.71	.97808	18.00	14.60	14.28	.97482	21.25	17.30	16.86
.98135	14.80	11.97	11.75	.97803	18.05	14.64	14.32	.97477	21.30	17.34	16.90
.98130	14.85	12.01	11.79	.97798	18.10	14.68	14.36	.97472	21.35	17.38	16.94
.98125	14.90	12.05	11.82	.97793	18.15	14.73	14.40	.97467	21.40	17.43	16.98
.98119	14.95	12.09	11.86	.97788	18.20	14.77	14.44	.97462	21.45	17.47	17.02
.98114	15.00	12.13	11.90	.97783	18.25	14.81	14.48	.97457	21.50	17.51	17.06
.98108	15.05	12.17	11.94	.97778	18.30	14.85	14.52	.97451	21.55	17.55	17.10
.98104	15.10	12.21	11.98	.97773	18.35	14.89	14.56	.97446	21.60	17.59	17.14
.98099	15.15	12.25	12.02	.97768	18.40	14.94	14.60	.97441	21.65	17.63	17.18
.98093	15.20	12.29	12.06	.97763	18.45	14.98	14.64	.97436	21.70	17.67	17.22

TABLE II.—*Percentage of alcohol*—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
0.97431	21.75	17.71	17.26	0.97097	25.00	20.43	19.84	0.96744	28.25	23.17	22.42
.97426	21.80	17.76	17.30	.97092	25.05	20.47	19.88	.96738	28.30	23.21	22.45
.97421	21.85	17.80	17.34	.97086	25.10	20.51	19.92	.96732	28.35	23.25	22.49
.97416	21.90	17.84	17.38	.97081	25.15	20.56	19.96	.96726	28.40	23.30	22.53
.97411	21.95	17.88	17.42	.97076	25.20	20.60	20.00	.96721	28.45	23.34	22.57
.97406	22.00	17.92	17.46	.97071	25.25	20.64	20.04	.96715	28.50	23.38	22.61
.97401	22.05	17.96	17.50	.97065	25.30	20.68	20.08	.96709	28.55	23.42	22.65
.97396	22.10	18.00	17.54	.97060	25.35	20.72	20.12	.96704	28.60	23.47	22.69
.97391	22.15	18.05	17.58	.97055	25.40	20.77	20.16	.96698	28.65	23.51	22.73
.97386	22.20	18.09	17.62	.97049	25.45	20.81	20.20	.96692	28.70	23.55	22.77
.97381	22.25	18.13	17.66	.97044	25.50	20.85	20.24	.96687	28.75	23.60	22.81
.97375	22.30	18.17	17.70	.97039	25.55	20.89	20.28	.96681	28.80	23.64	22.85
.97370	22.35	18.21	17.74	.97033	25.60	20.93	20.32	.96675	28.85	23.68	22.89
.97365	22.40	18.26	17.78	.97028	25.65	20.98	20.36	.96669	28.90	23.72	22.93
.97360	22.45	18.30	17.82	.97023	25.70	21.02	20.40	.96664	28.95	23.77	22.97
.97355	22.50	18.34	17.86	.97018	25.75	21.06	20.44	.96658	29.00	23.81	23.01
.97350	22.55	18.38	17.90	.97012	25.80	21.10	20.47	.96652	29.05	23.85	23.05
.97345	22.60	18.42	17.94	.97007	25.85	21.14	20.51	.96646	29.10	23.89	23.09
.97340	22.65	18.47	17.98	.97001	25.90	21.19	20.55	.96640	29.15	23.94	23.13
.97335	22.70	18.51	18.02	.96996	25.95	21.23	20.59	.96635	29.20	23.98	23.17
.97330	22.75	18.55	18.06	.96991	26.00	21.27	20.63	.96629	29.25	24.02	23.21
.97324	22.80	18.59	18.10	.96986	26.05	21.31	20.67	.96623	29.30	24.06	23.25
.97319	22.85	18.63	18.14	.96980	26.10	21.35	20.71	.96617	29.35	24.10	23.29
.97314	22.90	18.68	18.18	.96975	26.15	21.40	20.75	.96611	29.40	24.15	23.33
.97309	22.95	18.72	18.22	.96969	26.20	21.44	20.79	.96605	29.45	24.19	23.37
.97304	23.00	18.76	18.26	.96964	26.25	21.48	20.83	.96600	29.50	24.23	23.41
.97299	23.05	18.80	18.29	.96959	26.30	21.52	20.87	.96594	29.55	24.27	23.45
.97294	23.10	18.84	18.33	.96953	26.35	21.56	20.91	.96588	29.60	24.32	23.49
.97289	23.15	18.88	18.37	.96949	26.40	21.61	20.95	.96582	29.65	24.36	23.53
.97283	23.20	18.92	18.41	.96942	26.45	21.65	20.99	.96576	29.70	24.40	23.57
.97278	23.25	18.96	18.45	.96937	26.50	21.69	21.03	.96570	29.75	24.45	23.61
.97273	23.30	19.01	18.49	.96932	26.55	21.73	21.07	.96564	29.80	24.49	23.65
.97268	23.35	19.05	18.53	.96926	26.60	21.77	21.11	.96559	29.85	24.53	23.69
.97263	23.40	19.09	18.57	.96921	26.65	21.82	21.15	.96553	29.90	24.57	23.73
.97258	23.45	19.13	18.61	.96915	26.70	21.86	21.19	.96547	29.95	24.62	23.77
.97253	23.50	19.17	18.65	.96910	26.75	21.90	21.23	.96541	30.00	24.66	23.81
.97247	23.55	19.21	18.69	.96905	26.80	21.94	21.27	.96535	30.05	24.70	23.85
.97242	23.60	19.25	18.73	.96899	26.85	21.98	21.31	.96529	30.10	24.74	23.89
.97237	23.65	19.30	18.77	.96894	26.90	22.03	21.35	.96523	30.15	24.79	23.93
.97232	23.70	19.34	18.81	.96888	26.95	22.07	21.39	.96517	30.20	24.83	23.97
.97227	23.75	19.38	18.84	.96883	27.00	22.11	21.43	.96511	30.25	24.87	24.01
.97222	23.80	19.42	18.88	.96877	27.05	22.15	21.47	.96505	30.30	24.91	24.04
.97216	23.85	19.46	18.92	.96872	27.10	22.20	21.51	.96499	30.35	24.95	24.08
.97211	23.90	19.51	18.96	.96866	27.15	22.24	21.55	.96493	30.40	25.00	24.12
.97206	23.95	19.55	19.00	.96861	27.20	22.28	21.59	.96487	30.45	25.04	24.16
.97201	24.00	19.59	19.04	.96855	27.25	22.33	21.63	.96181	30.50	25.08	24.20
.97196	24.05	19.63	19.08	.96850	27.30	22.37	21.67	.96475	30.55	25.12	24.24
.97191	24.10	19.67	19.12	.96844	27.35	22.41	21.71	.96469	30.60	25.17	24.28
.97185	24.15	19.72	19.16	.96839	27.40	22.45	21.75	.96463	30.65	25.21	24.32
.97180	24.20	19.76	19.20	.96833	27.45	22.50	21.79	.96457	30.70	25.25	24.36
.97175	24.25	19.80	19.24	.96828	27.50	22.54	21.83	.96451	30.75	25.30	24.40
.97170	24.30	19.84	19.28	.96822	27.55	22.58	21.86	.96445	30.80	25.34	24.44
.97165	24.35	19.88	19.32	.96816	27.60	22.62	21.90	.96439	30.85	25.38	24.48
.97159	24.40	19.93	19.36	.96811	27.65	22.67	21.94	.96433	30.90	25.42	24.52
.97154	24.45	19.97	19.40	.96805	27.70	22.71	21.98	.96427	30.95	25.47	24.56
.97149	24.50	20.01	19.44	.96800	27.75	22.75	22.02	.96421	31.00	25.51	24.60
.97144	24.55	20.05	19.48	.96794	27.80	22.79	22.06	.96415	31.05	25.55	24.64
.97139	24.60	20.09	19.52	.96789	27.85	22.83	22.10	.96409	31.10	25.60	24.68
.97133	24.65	20.14	19.56	.96783	27.90	22.88	22.14	.96403	31.15	25.64	24.72
.97128	24.70	20.18	19.60	.96778	27.95	22.92	22.18	.96396	31.20	25.68	24.76
.97123	24.75	20.22	19.64	.96772	28.00	22.96	22.22	.96390	31.25	25.73	24.80
.97118	24.80	20.26	19.68	.96766	28.05	23.00	22.26	.96384	31.30	25.77	24.84
.97113	24.85	20.30	19.72	.96761	28.10	23.04	22.30	.96378	31.35	25.81	24.88
.97107	24.90	20.35	19.76	.96755	28.15	23.09	22.34	.96372	31.40	25.85	24.92
.97102	24.95	20.39	19.80	.96749	28.20	23.13	22.38	.96366	31.45	25.90	24.96

TABLE II. — *Percentage of alcohol*—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.		Per cent by volume.	Per cent by weight.	Grams per 100 cc.
.96360	31.50	25.94	25.00	.95943	34.75	28.74	27.58	.95487	38.00	31.58	30.16
.96353	31.55	25.98	25.04	.95937	34.80	28.78	27.62	.95480	38.05	31.63	30.20
.96347	31.60	26.03	25.08	.95930	34.85	28.83	27.66	.95472	38.10	31.67	30.24
.96341	31.65	26.07	25.12	.95923	34.90	28.87	27.70	.95465	38.15	31.72	30.28
.96335	31.70	26.11	25.16	.95917	34.95	28.92	27.74	.95457	38.20	31.76	30.32
.96329	31.75	26.16	25.20	.95910	35.00	28.96	27.78	.95450	38.25	31.81	30.36
.96323	31.80	26.20	25.24	.95903	35.05	29.00	27.82	.95442	38.30	31.85	30.40
.96316	31.85	26.24	25.28	.95896	35.10	29.05	27.86	.95435	38.35	31.90	30.44
.96310	31.90	26.28	25.32	.95889	35.15	29.09	27.90	.95427	38.40	31.94	30.48
.96304	31.95	26.33	25.36	.95883	35.20	29.13	27.94	.95420	38.45	31.99	30.52
.96298	32.00	26.37	25.40	.95876	35.25	29.18	27.98	.95413	38.50	32.03	30.56
.96292	32.05	26.41	25.44	.95869	35.30	29.22	28.02	.95405	38.55	32.07	30.60
.96285	32.10	26.46	25.48	.95862	35.35	29.26	28.05	.95398	38.60	32.12	30.64
.96279	32.15	26.50	25.52	.95855	35.40	29.30	28.09	.95390	38.65	32.16	30.68
.96273	32.20	26.54	25.56	.95848	35.45	29.35	28.13	.95383	38.70	32.20	30.72
.96267	32.25	26.59	25.60	.95842	35.50	29.40	28.17	.95375	38.75	32.25	30.76
.96260	32.30	26.63	25.64	.95835	35.55	29.43	28.21	.95368	38.80	32.29	30.79
.96254	32.35	26.67	25.68	.95828	35.60	29.48	28.25	.95360	38.85	32.33	30.83
.96248	32.40	26.71	25.71	.95821	35.65	29.52	28.29	.95353	38.90	32.37	30.87
.96241	32.45	26.76	25.75	.95814	35.70	29.57	28.33	.95345	38.95	32.42	30.91
.96235	32.50	26.80	25.79	.95807	35.75	29.61	28.37	.95338	39.00	32.46	30.95
.96229	32.55	26.84	25.83	.95800	35.80	29.65	28.41	.95330	39.05	32.50	30.99
.96222	32.60	26.89	25.87	.95794	35.85	29.70	28.45	.95323	39.10	32.55	31.03
.96216	32.65	26.93	25.91	.95787	35.90	29.74	28.49	.95315	39.15	32.59	31.07
.96210	32.70	26.97	25.95	.95780	35.95	29.79	28.53	.95307	39.20	32.64	31.11
.96204	32.75	27.02	25.99	.95773	36.00	29.83	28.57	.95300	39.25	32.68	31.14
.96197	32.80	27.06	26.03	.95766	36.05	29.87	28.61	.95292	39.30	32.72	31.18
.96191	32.85	27.10	26.07	.95759	36.10	29.92	28.65	.95284	39.35	32.77	31.22
.96185	32.90	27.14	26.11	.95752	36.15	29.96	28.69	.95277	39.40	32.81	31.26
.96178	32.95	27.19	26.15	.95745	36.20	30.00	28.73	.95269	39.45	32.86	31.30
.96172	33.00	27.23	26.19	.95738	36.25	30.05	28.77	.95262	39.50	32.90	31.34
.96166	33.05	27.27	26.23	.95731	36.30	30.09	28.81	.95254	39.55	32.95	31.38
.96159	33.10	27.32	26.27	.95724	36.35	30.13	28.84	.95246	39.60	32.99	31.42
.96153	33.15	27.36	26.31	.95717	36.40	30.17	28.88	.95239	39.65	33.04	31.46
.96146	33.20	27.40	26.35	.95710	36.45	30.22	28.92	.95231	39.70	33.08	31.50
.96140	33.25	27.45	26.39	.95703	36.50	30.26	28.96	.95223	39.75	33.13	31.54
.96133	33.30	27.49	26.43	.95695	36.55	30.30	29.00	.95216	39.80	33.17	31.58
.96127	33.35	27.53	26.47	.95688	36.60	30.35	29.04	.95208	39.85	33.22	31.62
.96120	33.40	27.57	26.51	.95681	36.65	30.39	29.08	.95200	39.90	33.27	31.66
.96114	33.45	27.62	26.55	.95674	36.70	30.44	29.12	.95193	39.95	33.31	31.70
.96108	33.50	27.66	26.59	.95667	36.75	30.48	29.16	.95185	40.00	33.35	31.74
.96101	33.55	27.70	26.63	.95660	36.80	30.52	29.20	.95177	40.05	33.39	31.78
.96095	33.60	27.75	26.67	.95653	36.85	30.57	29.24	.95169	40.10	33.44	31.82
.96088	33.65	27.79	26.71	.95646	36.90	30.61	29.29	.95161	40.15	33.48	31.86
.96082	33.70	27.83	26.75	.95639	36.95	30.66	29.32	.95154	40.20	33.53	31.90
.96075	33.75	27.88	26.79	.95632	37.00	30.70	29.36	.95146	40.25	33.57	31.94
.96069	33.80	27.92	26.82	.95625	37.05	30.74	29.40	.95138	40.30	33.61	31.98
.96062	33.85	27.96	26.86	.95618	37.10	30.79	29.44	.95130	40.35	33.66	32.02
.96056	33.90	28.00	26.90	.95610	37.15	30.83	29.48	.95122	40.40	33.70	32.06
.96049	33.95	28.05	26.94	.95603	37.20	30.88	29.52	.95114	40.45	33.75	32.10
.96043	34.00	28.09	26.98	.95596	37.25	30.92	29.56	.95107	40.50	33.79	32.14
.96036	34.05	28.13	27.02	.95589	37.30	30.96	29.60	.95099	40.55	33.84	32.18
.96030	34.10	28.18	27.06	.95581	37.35	31.01	29.64	.95091	40.60	33.88	32.22
.96023	34.15	28.22	27.10	.95574	37.40	31.05	29.68	.95083	40.65	33.93	32.26
.96016	34.20	28.26	27.14	.95567	37.45	31.10	29.72	.95075	40.70	33.97	32.30
.96010	34.25	28.31	27.18	.95560	37.50	31.14	29.76	.95067	40.75	34.02	32.34
.96003	34.30	28.35	27.22	.95552	37.55	31.18	29.80	.95059	40.80	34.06	32.38
.95996	34.35	28.39	27.26	.95545	37.60	31.23	29.84	.95052	40.85	34.11	32.42
.95990	34.40	28.43	27.30	.95538	37.65	31.27	29.88	.95044	40.90	34.15	32.46
.95983	34.45	28.48	27.34	.95531	37.70	31.32	29.92	.95036	40.95	34.20	32.50
.95977	34.50	28.52	27.38	.95523	37.75	31.36	29.96	.95028	41.00	34.24	32.54
.95970	34.55	28.56	27.42	.95516	37.80	31.40	30.00	.95020	41.05	34.28	32.58
.95963	34.60	28.61	27.46	.95509	37.85	31.45	30.04	.95012	41.10	34.33	32.62
.95957	34.65	28.65	27.50	.95502	37.90	31.49	30.08	.95004	41.15	34.37	32.66
.95950	34.70	28.70	27.54	.95494	37.95	31.54	30.12	.94996	41.20	34.42	32.70

TABLE II.—Percentage of alcohol—Continued.

Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.			Specific gravity at 60° F.	Alcohol.		
	Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.		Per cent by vol-ume.	Per cent by weight.	Grams per 100 cc.
0.94988	41.25	34.46	32.74	0.94498	44.25	37.16	35.11	0.93962	47.25	39.90	37.49
.94980	41.30	34.50	32.78	.94484	44.30	37.21	35.15	.93953	47.30	39.95	37.53
.94972	41.35	34.55	32.82	.94476	44.35	37.25	35.19	.93944	47.35	39.99	37.57
.94964	41.40	34.59	32.86	.94467	44.40	37.30	35.23	.93934	47.40	40.04	37.61
.94956	41.45	34.64	32.90	.94459	44.45	37.34	35.27	.93925	47.45	40.08	37.65
.94948	41.50	34.68	32.93	.94450	44.50	37.39	35.31	.93916	47.50	40.13	37.69
.94940	41.55	34.73	32.97	.94441	44.55	37.44	35.35	.93906	47.55	40.18	37.73
.94932	41.60	34.77	33.01	.94433	44.60	37.48	35.39	.93898	47.60	40.22	37.77
.94924	41.65	34.82	33.05	.94424	44.65	37.53	35.43	.93888	47.65	40.27	37.81
.94916	41.70	34.86	33.09	.94416	44.70	37.57	35.47	.93879	47.70	40.32	37.85
.94908	41.75	34.91	33.13	.94407	44.75	37.62	35.51	.93870	47.75	40.37	37.89
.94900	41.80	34.95	33.17	.94398	44.80	37.66	35.55	.93861	47.80	40.41	37.93
.94892	41.85	35.00	33.21	.94390	44.85	37.71	35.59	.93852	47.85	40.46	37.97
.94884	41.90	35.04	33.25	.94381	44.90	37.76	35.63	.93842	47.90	40.51	38.01
.94876	41.95	35.09	33.29	.94373	44.95	37.80	35.67	.93833	47.95	40.55	38.05
.94868	42.00	35.13	33.33	.94364	45.00	37.84	35.71	.93824	48.00	40.60	38.09
.94860	42.05	35.18	33.37	.94355	45.05	37.89	35.75	.93815	48.05	40.65	38.13
.94852	42.10	35.22	33.41	.94346	45.10	37.93	35.79	.93806	48.10	40.69	38.17
.94843	42.15	35.27	33.45	.94338	45.15	37.98	35.83	.93796	48.15	40.74	38.21
.94835	42.20	35.31	33.49	.94329	45.20	38.02	35.87	.93786	48.20	40.78	38.25
.94827	42.25	35.36	33.53	.94320	45.25	38.07	35.91	.93777	48.25	40.83	38.29
.94810	42.30	35.40	33.57	.94311	45.30	38.12	35.95	.93768	48.30	40.88	38.33
.94811	42.35	35.45	33.61	.94302	45.35	38.16	35.99	.93758	48.35	40.92	38.37
.94802	42.40	35.49	33.65	.94294	45.40	38.21	36.03	.93749	48.40	40.97	38.41
.94794	42.45	35.54	33.69	.94285	45.45	38.25	36.07	.93739	48.45	41.01	38.45
.94786	42.50	35.58	33.73	.94276	45.50	38.30	36.11	.93730	48.50	41.06	38.49
.94778	42.55	35.63	33.77	.94267	45.55	38.35	36.15	.93721	48.55	41.11	38.53
.94770	42.60	35.67	33.81	.94258	45.60	38.39	36.19	.93711	48.60	41.15	38.57
.94761	42.65	35.72	33.85	.94250	45.65	38.44	36.23	.93702	48.65	41.20	38.61
.94753	42.70	35.76	33.89	.94241	45.70	38.48	36.26	.93692	48.70	41.24	38.65
.94745	42.75	35.81	33.93	.94232	45.75	38.53	36.30	.93683	48.75	41.29	38.68
.94737	42.80	35.85	33.97	.94223	45.80	38.57	36.34	.93673	48.80	41.34	38.72
.94729	42.85	35.90	34.00	.94214	45.85	38.62	36.38	.93664	48.85	41.38	38.76
.94720	42.90	35.94	34.04	.94206	45.90	38.66	36.42	.93655	48.90	41.43	38.80
.94712	42.95	35.99	34.08	.94197	45.95	38.71	36.46	.93645	48.95	41.47	38.84
.94704	43.00	36.03	34.12	.94188	46.00	38.75	36.50	.93636	49.00	41.52	38.88
.94696	43.05	36.08	34.16	.94179	46.05	38.80	36.54	.93626	49.05	41.57	38.92
.94687	43.10	36.12	34.20	.94170	46.10	38.84	36.58	.93617	49.10	41.61	38.96
.94679	43.15	36.17	34.24	.94161	46.15	38.89	36.62	.93607	49.15	41.66	39.00
.94670	43.20	36.21	34.28	.94152	46.20	38.93	36.66	.93598	49.20	41.71	39.04
.94662	43.25	36.23	34.32	.94143	46.25	38.98	36.70	.93588	49.25	41.76	39.08
.94654	43.30	36.30	34.36	.94134	46.30	39.03	36.74	.93578	49.30	41.80	39.12
.94645	43.35	36.35	34.40	.94125	46.35	39.07	36.78	.93569	49.35	41.85	39.16
.94637	43.40	36.39	34.44	.94116	46.40	39.12	36.82	.93559	49.40	41.90	39.20
.94628	43.45	36.44	34.48	.94107	46.45	39.16	36.86	.93550	49.45	41.94	39.24
.94620	43.50	36.48	34.52	.94098	46.50	39.21	36.90	.93540	49.50	41.99	39.28
.94612	43.55	36.53	34.56	.94089	46.55	39.26	36.94	.93530	49.55	42.04	39.32
.94603	43.60	36.57	34.60	.94080	46.60	39.30	36.98	.93521	49.60	42.08	39.36
.94595	43.65	36.62	34.64	.94071	46.65	39.35	37.02	.93511	49.65	42.13	39.40
.94586	43.70	36.66	34.68	.94062	46.70	39.39	37.06	.93502	49.70	42.18	39.44
.94578	43.75	36.71	34.72	.94053	46.75	39.44	37.09	.93492	49.75	42.23	39.48
.94570	43.80	36.75	34.76	.94044	46.80	39.49	37.13	.93482	49.80	42.27	39.52
.94561	43.85	36.80	34.80	.94035	46.85	39.53	37.17	.93473	49.85	42.32	39.56
.94553	43.90	36.84	34.84	.94026	46.90	39.58	37.21	.93463	49.90	42.37	39.60
.94544	43.95	36.89	34.88	.94017	46.95	39.62	37.25	.93454	49.95	42.41	39.63
.94536	44.00	36.93	34.91	.94008	47.00	39.67	37.29				
.94527	44.05	36.98	34.95	.93999	47.05	39.72	37.33				
.94519	44.10	37.02	34.99	.93990	47.10	39.76	37.37				
.94510	44.15	37.07	35.03	.93980	47.15	39.81	37.41				
.94502	44.20	37.11	35.07	.93971	47.20	39.85	37.45				

TABLE III.—*Extract in beer wort.*^a

[According to Schultz and Ostermann.]

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0000	0.00	0.00	1.0065	1.69	1.70	1.0130	3.35	3.39	1.0195	5.06	5.16
1.0001	0.03	0.03	1.0066	1.72	1.73	1.0131	3.38	3.42	1.0196	5.09	5.19
1.0002	0.05	0.05	1.0067	1.74	1.75	1.0132	3.41	3.46	1.0197	5.12	5.22
1.0003	0.08	0.08	1.0068	1.77	1.78	1.0133	3.43	3.48	1.0198	5.15	5.25
1.0004	0.10	1.10	1.0069	1.79	1.80	1.0134	3.46	3.51	1.0199	5.17	5.27
1.0005	0.13	0.13	1.0070	1.82	1.83	1.0135	3.48	3.53	1.0200	5.20	5.30
1.0006	0.16	0.16	1.0071	1.84	1.85	1.0136	3.51	3.56	1.0201	5.23	5.34
1.0007	0.18	0.18	1.0072	1.87	1.88	1.0127	3.54	3.59	1.0202	5.25	5.36
1.0008	0.21	0.21	1.0073	1.90	1.91	1.0138	3.56	3.61	1.0203	5.28	5.39
1.0009	0.24	0.24	1.0074	1.92	1.93	1.0139	3.59	3.64	1.0204	5.30	5.41
1.0010	0.26	0.26	1.0075	1.95	1.96	1.0140	3.61	3.66	1.0205	5.33	5.44
1.0011	0.29	0.29	1.0076	1.97	1.98	1.0141	3.64	3.69	1.0206	5.35	5.46
1.0012	0.31	0.31	1.0077	2.00	2.02	1.0142	3.66	3.71	1.0207	5.38	5.49
1.0013	0.34	0.34	1.0078	2.02	2.04	1.0143	3.69	3.74	1.0208	5.40	5.51
1.0014	0.37	0.37	1.0079	2.05	2.07	1.0144	3.72	4.77	1.0209	5.43	5.54
1.0015	0.39	0.39	1.0080	2.07	2.09	1.0145	3.74	3.79	1.0210	5.45	5.56
1.0016	0.42	0.42	1.0081	2.10	2.12	1.0146	3.77	3.83	1.0211	5.48	5.60
1.0017	0.45	0.45	1.0082	2.12	2.14	1.0147	3.79	3.85	1.0212	5.50	5.62
1.0018	0.47	0.47	1.0083	2.15	2.17	1.0148	3.82	3.88	1.0213	5.53	5.65
1.0019	0.50	0.50	1.0084	2.17	2.19	1.0149	3.85	3.91	1.0214	5.55	5.67
1.0020	0.52	0.52	1.0085	2.20	2.22	1.0150	3.87	3.93	1.0215	5.57	5.69
1.0021	0.55	0.55	1.0086	2.23	2.25	1.0151	3.90	3.96	1.0216	5.60	5.72
1.0022	0.58	0.58	1.0087	2.25	2.27	1.0152	3.92	3.98	1.0217	5.62	5.74
1.0023	0.60	0.60	1.0088	2.28	2.30	1.0153	3.95	4.01	1.0218	5.65	5.77
1.0024	0.63	0.63	1.0089	2.30	2.32	1.0154	3.97	4.03	1.0219	5.67	5.79
1.0025	0.66	0.66	1.0090	2.33	2.35	1.0155	4.00	4.06	1.0220	5.70	5.83
1.0026	0.68	0.68	1.0091	2.35	2.37	1.0156	4.03	4.09	1.0221	5.72	5.85
1.0027	0.71	0.71	1.0092	2.38	2.40	1.0157	4.05	4.11	1.0222	5.75	5.88
1.0028	0.73	0.73	1.0093	2.41	2.43	1.0158	4.08	4.14	1.0223	5.77	5.90
1.0029	0.76	0.76	1.0094	2.43	2.45	1.0159	4.10	4.17	1.0224	5.80	5.93
1.0030	0.79	0.79	1.0095	2.46	2.48	1.0160	4.13	4.20	1.0225	5.82	5.95
1.0031	0.81	0.81	1.0096	2.48	2.50	1.0161	4.16	4.23	1.0226	5.84	5.97
1.0032	0.84	0.84	1.0097	2.51	2.53	1.0162	4.18	4.25	1.0227	5.87	6.00
1.0033	0.87	0.87	1.0098	2.53	2.55	1.0163	4.21	4.28	1.0228	5.89	6.02
1.0034	0.89	0.89	1.0099	2.56	2.59	1.0164	4.23	4.30	1.0229	5.92	6.06
1.0035	0.92	0.92	1.0100	2.58	2.61	1.0165	4.26	4.33	1.0230	5.94	6.08
1.0036	0.94	0.94	1.0101	2.61	2.64	1.0166	4.28	4.35	1.0231	5.97	6.11
1.0037	0.97	0.97	1.0102	2.64	2.67	1.0167	4.31	4.38	1.0232	5.99	6.13
1.0038	1.00	1.00	1.0103	2.66	2.69	1.0168	4.34	4.41	1.0233	6.02	6.16
1.0039	1.02	1.02	1.0104	2.69	2.72	1.0169	4.36	4.43	1.0234	6.04	6.18
1.0040	1.05	1.05	1.0105	2.71	2.74	1.0170	4.39	4.46	1.0235	6.07	6.21
1.0041	1.08	1.08	1.0106	2.74	2.77	1.0171	4.42	4.50	1.0236	6.09	6.23
1.0042	1.10	1.10	1.0107	2.76	2.79	1.0172	4.44	4.52	1.0237	6.11	6.25
1.0043	1.13	1.13	1.0108	2.79	2.82	1.0173	4.47	4.55	1.0238	6.14	6.29
1.0044	1.15	1.16	1.0109	2.82	2.85	1.0174	4.50	4.58	1.0239	6.16	6.31
1.0045	1.18	1.19	1.0110	2.84	2.87	1.0175	4.53	4.61	1.0240	6.19	6.34
1.0046	1.21	1.22	1.0111	2.87	2.90	1.0176	4.55	4.63	1.0241	6.21	6.36
1.0047	1.23	1.24	1.0112	2.89	2.92	1.0177	4.58	4.66	1.0242	6.24	6.39
1.0048	1.26	1.27	1.0113	2.92	2.95	1.0178	4.61	4.69	1.0243	6.26	6.41
1.0049	1.29	1.30	1.0114	2.94	2.97	1.0179	4.63	4.71	1.0244	6.29	6.44
1.0050	1.31	1.32	1.0115	2.97	3.00	1.0180	4.66	4.74	1.0245	6.31	6.46
1.0051	1.34	1.35	1.0116	2.99	3.02	1.0181	4.69	4.77	1.0246	6.34	6.50
1.0052	1.36	1.37	1.0117	3.02	3.06	1.0182	4.71	4.80	1.0247	6.36	6.52
1.0053	1.39	1.40	1.0118	3.05	3.09	1.0183	4.74	4.83	1.0248	6.39	6.55
1.0054	1.41	1.42	1.0119	3.07	3.11	1.0184	4.77	4.86	1.0249	6.41	6.57
1.0055	1.44	1.45	1.0120	3.10	3.14	1.0185	4.79	4.88	1.0250	6.44	6.60
1.0056	1.46	1.47	1.0121	3.12	3.16	1.0186	4.82	4.91	1.0251	6.47	6.63
1.0057	1.49	1.50	1.0122	3.15	3.19	1.0187	4.85	4.94	1.0252	6.50	6.66
1.0058	1.51	1.52	1.0123	3.17	3.21	1.0188	4.88	4.97	1.0253	6.52	6.68
1.0059	1.54	1.55	1.0124	3.20	3.24	1.0189	4.90	4.99	1.0254	6.55	6.72
1.0060	1.56	1.57	1.0125	3.23	3.27	1.0190	4.93	5.02	1.0255	6.58	6.75
1.0061	1.59	1.60	1.0126	3.25	3.29	1.0191	4.96	5.05	1.0256	6.61	6.78
1.0062	1.62	1.63	1.0127	3.28	3.32	1.0192	4.98	5.08	1.0257	6.63	6.80
1.0063	1.64	1.65	1.0128	3.30	3.34	1.0193	5.01	5.11	1.0258	6.66	6.83
1.0064	1.67	1.68	1.0129	3.33	3.37	1.0194	5.04	5.14	1.0259	6.69	6.86

^a Calculated from results obtained by drying below 75° C.

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0260	6.71	6.88	1.0325	8.27	8.54	1.0390	9.92	10.31	1.0455	11.53	12.05
1.0261	6.74	6.92	1.0326	8.29	8.56	1.0391	9.95	10.34	1.0456	11.55	12.08
1.0262	6.77	6.95	1.0327	8.32	8.59	1.0392	9.97	10.36	1.0457	11.57	12.10
1.0263	6.80	6.98	1.0328	8.34	8.61	1.0393	9.99	10.38	1.0458	11.60	12.13
1.0264	6.82	7.00	1.0329	8.37	8.65	1.0394	10.02	10.41	1.0459	11.62	12.15
1.0265	6.85	7.03	1.0330	8.40	8.68	1.0395	10.04	10.44	1.0460	11.65	12.19
1.0266	6.88	7.06	1.0331	8.43	8.71	1.0396	10.06	10.46	1.0461	11.67	12.21
1.0267	6.91	7.09	1.0332	8.45	8.73	1.0397	10.09	10.49	1.0462	11.70	12.24
1.0268	6.93	7.12	1.0333	8.48	8.76	1.0398	10.11	10.51	1.0463	11.72	12.26
1.0269	6.96	7.15	1.0334	8.51	8.79	1.0399	10.13	10.53	1.0464	11.75	12.30
1.0270	6.99	7.18	1.0335	8.53	8.82	1.0400	10.16	10.57	1.0465	11.77	12.32
1.0271	7.01	7.20	1.0336	8.56	8.85	1.0401	10.18	10.59	1.0466	11.79	12.34
1.0272	7.04	7.23	1.0337	8.59	8.88	1.0402	10.20	10.61	1.0467	11.82	12.37
1.0273	7.07	7.26	1.0338	8.61	8.90	1.0403	10.23	10.64	1.0468	11.84	12.39
1.0274	7.10	7.29	1.0339	8.64	8.93	1.0404	10.25	10.66	1.0469	11.87	12.43
1.0275	7.12	7.32	1.0340	8.67	8.96	1.0405	10.27	10.69	1.0470	11.89	12.45
1.0276	7.15	7.35	1.0341	8.70	9.00	1.0406	10.30	10.72	1.0471	11.92	12.48
1.0277	7.18	7.38	1.0342	8.72	9.02	1.0407	10.32	10.74	1.0472	11.94	12.50
1.0278	7.21	7.41	1.0343	8.75	9.05	1.0408	10.35	10.77	1.0473	11.97	12.54
1.0279	7.23	7.43	1.0344	8.78	9.08	1.0409	10.37	10.79	1.0474	11.99	12.56
1.0280	7.26	7.46	1.0345	8.80	9.10	1.0410	10.40	10.83	1.0475	12.01	12.58
1.0281	7.28	7.48	1.0346	8.83	9.14	1.0411	10.42	10.85	1.0476	12.04	12.61
1.0282	7.30	7.51	1.0347	8.86	9.17	1.0412	10.45	10.88	1.0477	12.06	12.64
1.0283	7.33	7.54	1.0348	8.88	9.19	1.0413	10.47	10.90	1.0478	12.09	12.67
1.0284	7.35	7.56	1.0349	8.91	9.22	1.0414	10.50	10.93	1.0479	12.11	12.69
1.0285	7.37	7.58	1.0350	8.94	9.25	1.0415	10.52	10.96	1.0480	12.14	12.72
1.0286	7.39	7.60	1.0351	8.97	9.28	1.0416	10.55	10.99	1.0481	12.16	12.74
1.0287	7.42	7.63	1.0352	8.99	9.31	1.0417	10.57	11.01	1.0482	12.19	12.78
1.0288	7.44	7.65	1.0353	9.02	9.34	1.0418	10.60	11.04	1.0483	12.21	12.80
1.0289	7.46	7.68	1.0354	9.05	9.37	1.0419	10.62	11.06	1.0484	12.23	12.82
1.0290	7.48	7.70	1.0355	9.07	9.39	1.0420	10.65	11.10	1.0485	12.26	12.85
1.0291	7.51	7.73	1.0356	9.10	9.42	1.0421	10.67	11.12	1.0486	12.28	12.88
1.0292	7.53	7.75	1.0357	9.13	9.46	1.0422	10.70	11.15	1.0487	12.31	12.91
1.0293	7.55	7.77	1.0358	9.15	9.48	1.0423	10.72	11.17	1.0488	12.33	12.93
1.0294	7.57	7.79	1.0359	9.18	9.51	1.0424	10.75	11.21	1.0489	12.36	12.96
1.0295	7.60	7.82	1.0360	9.21	9.54	1.0425	10.77	11.23	1.0490	12.38	12.99
1.0296	7.62	7.85	1.0361	9.24	9.57	1.0426	10.80	11.26	1.0491	12.41	13.02
1.0297	7.64	7.87	1.0362	9.26	9.60	1.0427	10.82	11.28	1.0492	12.43	13.04
1.0298	7.66	7.89	1.0363	9.29	9.63	1.0428	10.85	11.31	1.0493	12.45	13.06
1.0299	7.69	7.92	1.0364	9.31	9.65	1.0429	10.88	11.35	1.0494	12.48	13.10
1.0300	7.71	7.94	1.0365	9.34	9.68	1.0430	10.90	11.37	1.0495	12.50	13.12
1.0301	7.73	7.96	1.0366	9.36	9.70	1.0431	10.93	11.40	1.0496	12.53	13.15
1.0302	7.75	7.98	1.0367	9.38	9.72	1.0432	10.95	11.42	1.0497	12.55	13.17
1.0303	7.77	8.01	1.0368	9.41	9.76	1.0433	10.98	11.46	1.0498	12.58	13.21
1.0304	7.80	8.04	1.0369	9.43	9.78	1.0434	11.00	11.48	1.0499	12.60	13.23
1.0305	7.82	8.06	1.0370	9.45	9.80	1.0435	11.03	11.51	1.0500	12.63	13.26
1.0306	7.84	8.08	1.0371	9.48	9.83	1.0436	11.05	11.53	1.0501	12.65	13.28
1.0307	7.86	8.10	1.0372	9.50	9.85	1.0437	11.08	11.56	1.0502	12.67	13.31
1.0308	7.89	8.13	1.0373	9.52	9.88	1.0438	11.10	11.59	1.0503	12.70	13.34
1.0309	7.91	8.15	1.0374	9.55	9.91	1.0439	11.13	11.62	1.0504	12.72	13.36
1.0310	7.93	8.18	1.0375	9.57	9.93	1.0440	11.15	11.64	1.0505	12.75	13.39
1.0311	7.95	8.20	1.0376	9.59	9.95	1.0441	11.18	11.67	1.0506	12.77	13.42
1.0312	7.98	8.23	1.0377	9.62	9.98	1.0442	11.20	11.70	1.0507	12.80	13.45
1.0313	8.00	8.25	1.0378	9.64	10.00	1.0443	11.23	11.73	1.0508	12.82	13.47
1.0314	8.02	8.27	1.0379	9.66	10.03	1.0444	11.25	11.75	1.0509	12.85	13.50
1.0315	8.04	8.29	1.0380	9.69	10.06	1.0445	11.28	11.78	1.0510	12.87	13.53
1.0316	8.07	8.33	1.0381	9.71	10.08	1.0446	11.30	11.80	1.0511	12.90	13.56
1.0317	8.09	8.35	1.0382	9.73	10.10	1.0447	11.33	11.84	1.0512	12.92	13.58
1.0318	8.11	8.37	1.0383	9.76	10.13	1.0448	11.35	11.86	1.0513	12.94	13.60
1.0319	8.13	8.39	1.0384	9.78	10.16	1.0449	11.38	11.89	1.0514	12.97	13.64
1.0320	8.16	8.42	1.0385	9.81	10.19	1.0450	11.40	11.91	1.0515	12.99	13.66
1.0321	8.18	8.44	1.0386	9.83	10.21	1.0451	11.43	11.95	1.0516	12.92	13.69
1.0322	8.20	8.46	1.0387	9.85	10.23	1.0452	11.45	11.97	1.0517	12.94	13.71
1.0323	8.22	8.49	1.0388	9.88	10.26	1.0453	11.48	12.00	1.0518	12.97	13.75
1.0324	8.25	8.52	1.0389	9.90	10.29	1.0454	11.50	12.02	1.0519	12.99	13.77

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0520	13.12	13.80	1.0585	14.75	15.61	1.0650	16.25	17.31	1.0715	17.81	19.08
1.0521	13.14	13.82	1.0586	14.78	15.65	1.0651	16.27	17.33	1.0716	17.84	19.12
1.0522	13.16	13.85	1.0587	14.81	15.68	1.0652	16.30	17.36	1.0717	17.86	19.14
1.0523	13.19	13.88	1.0588	14.83	15.70	1.0653	16.32	17.39	1.0718	17.88	19.16
1.0524	13.21	13.90	1.0589	14.86	15.74	1.0654	16.35	17.42	1.0719	17.90	19.19
1.0525	13.24	13.94	1.0590	14.89	15.77	1.0655	16.37	17.44	1.0720	17.93	19.22
1.0526	13.26	13.96	1.0591	14.91	15.79	1.0656	16.40	17.48	1.0721	17.95	19.24
1.0527	13.29	13.99	1.0592	14.94	15.82	1.0657	16.42	17.50	1.0722	17.97	19.27
1.0528	13.31	14.01	1.0593	14.96	15.85	1.0658	16.45	17.53	1.0723	17.99	19.29
1.0529	13.34	14.05	1.0594	14.99	15.88	1.0659	16.47	17.56	1.0724	18.02	19.32
1.0530	13.36	14.07	1.0595	15.02	15.91	1.0660	16.50	17.59	1.0725	18.04	19.35
1.0531	13.38	14.09	1.0596	15.04	15.94	1.0661	16.52	17.61	1.0726	18.06	19.37
1.0532	13.41	14.12	1.0597	15.07	15.97	1.0662	16.54	17.63	1.0727	18.08	19.39
1.0533	13.43	14.15	1.0598	15.09	15.99	1.0663	16.57	17.67	1.0728	18.11	19.43
1.0534	13.46	14.18	1.0599	15.11	16.02	1.0664	16.59	17.69	1.0729	18.13	19.45
1.0535	13.48	14.20	1.0600	15.14	16.05	1.0665	16.62	17.73	1.0730	18.15	19.47
1.0536	13.51	14.23	1.0601	15.16	16.07	1.0666	16.64	17.75	1.0731	18.17	19.50
1.0537	13.53	14.26	1.0602	15.18	16.09	1.0667	16.67	17.78	1.0732	18.20	19.53
1.0538	13.56	14.29	1.0603	15.20	16.12	1.0668	16.69	17.80	1.0733	18.22	19.55
1.0539	13.58	14.31	1.0604	15.23	16.15	1.0669	16.72	17.84	1.0734	18.24	19.58
1.0540	13.61	14.34	1.0605	15.25	16.17	1.0670	16.74	17.86	1.0735	18.26	19.60
1.0541	13.63	14.37	1.0606	15.27	16.20	1.0671	16.76	17.88	1.0736	18.29	19.64
1.0542	13.66	14.40	1.0607	15.29	16.22	1.0672	16.79	17.92	1.0737	18.31	19.66
1.0543	13.68	14.42	1.0608	15.31	16.24	1.0673	16.81	17.94	1.0738	18.33	19.68
1.0544	13.71	14.46	1.0609	15.34	16.27	1.0674	16.84	17.98	1.0739	18.35	19.71
1.0545	13.73	14.48	1.0610	15.36	16.30	1.0675	16.86	18.00	1.0740	18.38	19.74
1.0546	13.76	14.51	1.0611	15.38	16.32	1.0676	16.89	18.03	1.0741	18.40	19.76
1.0547	13.78	14.53	1.0612	15.40	16.34	1.0677	16.91	18.05	1.0742	18.42	19.79
1.0548	13.81	14.57	1.0613	15.43	16.38	1.0678	16.94	18.09	1.0743	18.44	19.81
1.0549	13.83	14.59	1.0614	15.45	16.40	1.0679	16.96	18.11	1.0744	18.47	19.84
1.0550	13.86	14.62	1.0615	15.47	16.42	1.0680	16.99	18.15	1.0745	18.49	19.87
1.0551	13.88	14.64	1.0616	15.49	16.44	1.0681	17.01	18.17	1.0746	18.51	19.89
1.0552	13.91	14.68	1.0617	15.52	16.48	1.0682	17.03	18.19	1.0747	18.53	19.91
1.0553	13.93	14.70	1.0618	15.54	16.50	1.0683	17.06	18.23	1.0748	18.55	19.94
1.0554	13.96	14.73	1.0619	15.56	16.52	1.0684	17.08	18.25	1.0749	18.57	19.96
1.0555	13.98	14.76	1.0620	15.58	16.55	1.0685	17.11	18.28	1.0750	18.59	19.98
1.0556	14.01	14.79	1.0621	15.60	16.57	1.0686	17.13	18.31	1.0751	18.62	20.02
1.0557	14.03	14.81	1.0622	15.63	16.60	1.0687	17.16	18.34	1.0752	18.64	20.04
1.0558	14.06	14.84	1.0623	15.65	16.62	1.0688	17.18	18.36	1.0753	18.66	20.07
1.0559	14.08	14.87	1.0624	15.67	16.64	1.0689	17.21	18.40	1.0754	18.68	20.09
1.0560	14.11	14.90	1.0625	15.69	16.66	1.0690	17.23	18.42	1.0755	18.70	20.11
1.0561	14.13	14.92	1.0626	15.72	16.70	1.0691	17.25	18.44	1.0756	18.72	20.14
1.0562	14.16	14.96	1.0627	15.74	16.73	1.0692	17.28	18.48	1.0757	18.74	20.16
1.0563	14.18	14.98	1.0628	15.76	16.75	1.0693	17.30	18.50	1.0758	18.76	20.18
1.0564	14.21	15.01	1.0629	15.78	16.77	1.0694	17.33	18.53	1.0759	18.78	20.21
1.0565	14.23	15.03	1.0630	15.80	16.80	1.0695	17.35	18.56	1.0760	18.81	20.24
1.0566	14.26	15.07	1.0631	15.83	16.83	1.0696	17.38	18.59	1.0761	18.83	20.26
1.0567	14.28	15.09	1.0632	15.85	16.85	1.0697	17.40	18.61	1.0762	18.85	20.29
1.0568	14.31	15.12	1.0633	15.87	16.87	1.0698	17.43	18.65	1.0763	18.87	20.31
1.0569	14.33	15.15	1.0634	15.89	16.90	1.0699	17.45	18.67	1.0764	18.89	20.33
1.0570	14.36	15.18	1.0635	15.92	16.93	1.0700	17.48	18.70	1.0765	18.91	20.36
1.0571	14.38	15.20	1.0636	15.94	16.95	1.0701	17.50	18.73	1.0766	18.93	20.38
1.0572	14.41	15.23	1.0637	15.96	16.98	1.0702	17.52	18.75	1.0767	18.95	20.40
1.0573	14.44	15.27	1.0638	15.98	17.00	1.0703	17.54	18.77	1.0768	18.97	20.43
1.0574	14.46	15.29	1.0639	16.01	17.03	1.0704	17.57	18.81	1.0769	19.00	20.46
1.0575	14.49	15.32	1.0640	16.03	17.06	1.0705	17.59	18.83	1.0770	19.02	20.48
1.0576	14.52	15.36	1.0641	16.05	17.08	1.0706	17.61	18.85	1.0771	19.04	20.51
1.0577	14.54	15.38	1.0642	16.07	17.10	1.0707	17.63	18.88	1.0772	19.06	20.53
1.0578	14.57	15.41	1.0643	16.09	17.12	1.0708	17.66	18.91	1.0773	19.08	20.55
1.0579	14.59	15.43	1.0644	16.12	17.16	1.0709	17.68	18.93	1.0774	19.10	20.58
1.0580	14.62	15.47	1.0645	16.14	17.18	1.0710	17.70	18.96	1.0775	19.12	20.60
1.0581	14.65	15.50	1.0646	16.16	17.20	1.0711	17.72	18.98	1.0776	19.14	20.63
1.0582	14.67	15.52	1.0647	16.18	17.23	1.0712	17.75	19.01	1.0777	19.17	20.66
1.0583	14.70	15.56	1.0648	16.21	17.26	1.0713	17.77	19.04	1.0778	19.19	20.68
1.0584	14.73	15.59	1.0649	16.23	17.28	1.0714	17.79	19.06	1.0779	19.21	20.71

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.		Specific gravity at 15°C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0780	19.23	20.73	1.0845	20.70	22.45	1.0910	22.19	24.21	1.0975	23.59	25.89
1.0781	19.25	20.75	1.0846	20.73	22.48	1.0911	22.21	24.24	1.0976	23.61	25.92
1.0782	19.27	20.78	1.0847	20.75	22.50	1.0912	22.23	24.26	1.0977	23.63	25.94
1.0783	19.29	20.80	1.0848	20.77	22.53	1.0913	22.26	24.29	1.0978	23.65	25.97
1.0784	19.31	20.82	1.0849	20.79	22.55	1.0914	22.28	24.31	1.0979	23.67	25.99
1.0785	19.33	20.85	1.0850	20.81	22.58	1.0915	22.30	24.34	1.0980	23.69	26.01
1.0786	19.36	20.88	1.0851	20.83	22.61	1.0916	22.32	24.37	1.0981	23.71	26.04
1.0787	19.38	20.90	1.0852	20.86	22.64	1.0917	22.34	24.39	1.0982	23.73	26.06
1.0788	19.40	20.93	1.0853	20.88	22.66	1.0918	22.37	24.42	1.0983	23.76	26.09
1.0789	19.42	20.95	1.0854	20.90	22.68	1.0919	22.39	24.44	1.0984	23.78	26.11
1.0790	19.44	20.98	1.0855	20.93	22.72	1.0920	22.41	24.47	1.0985	23.80	26.14
1.0791	19.46	21.00	1.0856	20.95	22.75	1.0921	22.43	24.49	1.0986	23.82	26.17
1.0792	19.49	21.03	1.0857	20.98	22.78	1.0922	22.45	24.51	1.0987	23.84	26.19
1.0793	19.51	21.06	1.0858	21.01	22.81	1.0923	22.48	24.54	1.0988	23.86	26.22
1.0794	19.53	21.08	1.0859	21.04	22.84	1.0924	22.50	24.56	1.0989	23.88	26.24
1.0795	19.56	21.11	1.0860	21.06	22.87	1.0925	22.52	24.60	1.0990	23.90	26.27
1.0796	19.58	21.14	1.0861	21.09	22.90	1.0926	22.54	24.62	1.0991	23.92	26.30
1.0797	19.60	21.16	1.0862	21.11	22.93	1.0927	22.56	24.64	1.0992	23.94	26.32
1.0798	19.63	21.20	1.0863	21.13	22.96	1.0928	22.59	24.67	1.0993	23.97	26.35
1.0799	19.65	21.22	1.0864	21.16	22.99	1.0929	22.61	24.70	1.0994	23.99	26.37
1.0800	19.67	21.24	1.0865	21.19	23.02	1.0930	22.63	24.73	1.0995	24.01	26.40
1.0801	19.70	21.28	1.0866	21.22	23.06	1.0931	22.65	24.76	1.0996	24.03	26.42
1.0802	19.72	21.30	1.0867	21.25	23.09	1.0932	22.67	24.78	1.0997	24.05	26.44
1.0803	19.74	21.33	1.0868	21.28	23.12	1.0933	22.69	24.81	1.0998	24.07	26.47
1.0804	19.77	21.36	1.0869	21.30	23.15	1.0934	22.71	24.83	1.0999	24.09	26.49
1.0805	19.79	21.38	1.0870	21.33	23.18	1.0935	22.73	24.86	1.1000	24.11	26.52
1.0806	19.81	21.41	1.0871	21.35	23.21	1.0936	22.75	24.89	1.1001	24.13	26.55
1.0807	19.84	21.43	1.0872	21.37	23.23	1.0937	22.77	24.91	1.1002	24.15	26.57
1.0808	19.86	21.46	1.0873	21.39	23.26	1.0938	22.80	24.93	1.1003	24.17	26.60
1.0809	19.88	21.49	1.0874	21.41	23.28	1.0939	22.82	24.96	1.1004	24.19	26.62
1.0810	19.91	21.52	1.0875	21.43	23.31	1.0940	22.84	24.99	1.1005	24.21	26.65
1.0811	19.93	21.55	1.0876	21.45	23.33	1.0941	22.86	25.01	1.1006	24.23	26.68
1.0812	19.96	21.58	1.0877	21.47	23.36	1.0942	22.88	25.03	1.1007	24.25	26.70
1.0813	19.98	21.60	1.0878	21.49	23.38	1.0943	22.90	25.06	1.1008	24.28	26.73
1.0814	20.00	21.63	1.0879	21.51	23.40	1.0944	22.92	25.08	1.1009	24.30	26.75
1.0815	20.03	21.66	1.0880	21.54	23.43	1.0945	22.94	25.11	1.1010	24.32	26.78
1.0816	20.05	21.69	1.0881	21.56	23.45	1.0946	22.96	25.14	1.1011	24.34	26.81
1.0817	20.07	21.71	1.0882	21.58	23.48	1.0947	22.98	25.16	1.1012	24.36	26.83
1.0818	20.10	21.74	1.0883	21.60	23.50	1.0948	23.00	25.18	1.1013	24.39	26.86
1.0819	20.12	21.77	1.0884	21.62	23.52	1.0949	23.03	25.21	1.1014	24.41	26.88
1.0820	20.14	21.79	1.0885	21.64	23.55	1.0950	23.05	25.24	1.1015	24.43	26.91
1.0821	20.17	21.83	1.0886	21.66	23.58	1.0951	23.07	25.26	1.1016	24.45	26.93
1.0822	20.19	21.85	1.0887	21.68	23.60	1.0952	23.10	25.29	1.1017	24.47	26.95
1.0823	20.21	21.87	1.0888	21.71	23.63	1.0953	23.12	25.31	1.1018	24.49	26.98
1.0824	20.24	21.91	1.0889	21.73	23.66	1.0954	23.14	25.34	1.1019	24.51	27.00
1.0825	20.26	21.93	1.0890	21.75	23.69	1.0955	23.16	25.37	1.1020	24.53	27.03
1.0826	20.28	21.96	1.0891	21.77	23.72	1.0956	23.18	25.39	1.1021	24.55	27.06
1.0827	20.31	21.99	1.0892	21.79	23.74	1.0957	23.20	25.42	1.1022	24.57	27.08
1.0828	20.33	22.01	1.0893	21.82	23.77	1.0958	23.23	25.45	1.1023	24.60	27.11
1.0829	20.35	22.04	1.0894	21.84	23.79	1.0959	23.25	25.47	1.1024	24.62	27.14
1.0830	20.37	22.06	1.0895	21.86	23.82	1.0960	23.27	25.50	1.1025	24.64	27.17
1.0831	20.39	22.08	1.0896	21.89	23.85	1.0961	23.29	25.53	1.1026	24.66	27.19
1.0832	20.41	22.11	1.0897	21.91	23.87	1.0962	23.31	25.55	1.1027	24.68	27.21
1.0833	20.43	22.13	1.0898	21.93	23.90	1.0963	23.33	25.58	1.1028	24.70	27.24
1.0834	20.46	22.16	1.0899	21.96	23.93	1.0964	23.35	25.60	1.1029	24.72	27.26
1.0835	20.48	22.19	1.0900	21.98	23.96	1.0965	23.37	25.63	1.1030	24.74	27.29
1.0836	20.50	22.21	1.0901	22.00	23.98	1.0966	23.39	25.66	1.1031	24.76	27.32
1.0837	20.52	22.24	1.0902	22.02	24.01	1.0967	23.41	25.68	1.1032	24.78	27.34
1.0838	20.54	22.26	1.0903	22.04	24.03	1.0968	23.44	25.71	1.1033	24.81	27.37
1.0839	20.56	22.29	1.0904	22.06	24.05	1.0969	23.46	25.73	1.1034	24.83	27.39
1.0840	20.59	22.32	1.0905	22.08	24.08	1.0970	23.48	25.76	1.1035	24.85	27.42
1.0841	20.62	22.35	1.0906	22.10	24.11	1.0971	23.50	25.79	1.1036	24.87	27.45
1.0842	20.64	22.38	1.0907	22.12	24.13	1.0972	23.52	25.81	1.1037	24.89	27.47
1.0843	20.66	22.40	1.0908	22.15	24.16	1.0973	23.55	25.84	1.1038	24.92	27.50
1.0844	20.68	22.42	1.0909	22.17	24.18	1.0974	23.57	25.86	1.1039	24.94	27.53

TABLE III.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.1040	24.96	27.56	1.1095	26.16	29.03	1.1150	27.29	30.43	1.1205	28.38	31.81
1.1041	24.98	27.58	1.1096	26.18	29.06	1.1151	27.31	30.45	1.1206	28.40	31.83
1.1042	25.00	27.60	1.1097	26.20	29.08	1.1152	27.33	30.47	1.1207	28.42	31.86
1.1043	25.03	27.63	1.1098	26.23	29.11	1.1153	27.35	30.50	1.1208	28.44	31.88
1.1044	25.05	27.66	1.1099	26.25	29.13	1.1154	27.37	30.52	1.1209	28.46	31.90
1.1045	25.07	27.69	1.1100	26.27	29.16	1.1155	27.38	30.55	1.1210	28.48	31.93
1.1046	25.09	27.72	1.1101	26.29	29.19	1.1156	27.40	30.57	1.1211	28.50	31.95
1.1047	25.11	27.74	1.1102	26.31	29.21	1.1157	27.42	30.59	1.1212	28.52	31.98
1.1048	25.14	27.77	1.1103	26.33	29.24	1.1158	27.44	30.62	1.1213	28.54	32.00
1.1049	25.16	27.79	1.1104	26.35	29.26	1.1159	27.46	30.64	1.1214	28.56	32.03
1.1050	25.18	27.82	1.1105	26.37	29.29	1.1160	27.48	30.67	1.1215	28.58	32.05
1.1051	25.20	27.85	1.1106	26.39	29.32	1.1161	27.50	30.69	1.1216	28.60	32.08
1.1052	25.22	27.87	1.1107	26.41	29.34	1.1162	27.52	30.72	1.1217	28.62	32.11
1.1053	25.24	27.90	1.1108	26.44	29.37	1.1163	27.54	30.75	1.1218	28.64	32.13
1.1054	25.27	27.93	1.1109	26.46	29.39	1.1164	27.56	30.77	1.1219	28.66	32.15
1.1055	25.29	27.96	1.1110	26.48	29.42	1.1165	27.58	30.80	1.1220	28.68	32.18
1.1056	25.31	27.98	1.1111	26.50	29.44	1.1166	27.60	30.82	1.1221	28.70	32.20
1.1057	25.33	28.00	1.1112	26.52	29.46	1.1167	27.62	30.85	1.1222	28.72	32.23
1.1058	25.35	28.03	1.1113	26.54	29.49	1.1168	27.64	30.87	1.1223	28.74	32.25
1.1059	25.38	28.06	1.1114	26.56	29.51	1.1169	27.66	30.89	1.1224	28.76	32.27
1.1060	25.40	28.09	1.1115	26.58	29.54	1.1170	27.68	30.92	1.1225	28.78	32.30
1.1061	25.42	28.12	1.1116	26.60	29.57	1.1171	27.70	30.94	1.1226	28.80	32.32
1.1062	25.44	28.14	1.1117	26.62	29.59	1.1172	27.72	30.97	1.1227	28.82	32.35
1.1063	25.46	28.17	1.1118	26.64	29.61	1.1173	27.74	31.00	1.1228	28.84	32.37
1.1064	25.48	28.19	1.1119	26.66	29.64	1.1174	27.76	31.02	1.1229	28.86	32.40
1.1065	25.50	28.22	1.1120	26.68	29.67	1.1175	27.78	31.05	1.1230	28.88	32.43
1.1066	25.52	28.25	1.1121	26.70	29.69	1.1176	27.80	31.07	1.1231	28.90	32.45
1.1067	25.54	28.27	1.1122	26.72	29.71	1.1177	27.82	31.09	1.1232	28.92	32.48
1.1068	25.57	28.30	1.1123	26.75	29.74	1.1178	27.84	31.12	1.1233	28.94	32.50
1.1069	25.59	28.32	1.1124	26.77	29.77	1.1179	27.86	31.15	1.1234	28.96	32.53
1.1070	25.61	28.35	1.1125	26.79	29.80	1.1180	27.88	31.18	1.1235	28.98	32.56
1.1071	25.63	28.38	1.1126	26.81	29.83	1.1181	27.90	31.20	1.1236	29.00	32.58
1.1072	25.65	28.40	1.1127	26.83	29.85	1.1182	27.92	31.23	1.1237	29.02	32.60
1.1073	25.67	28.43	1.1128	26.85	29.88	1.1183	27.94	31.25	1.1238	29.04	32.63
1.1074	25.69	28.45	1.1129	26.87	29.90	1.1184	27.96	31.27	1.1239	29.06	32.65
1.1075	25.71	28.48	1.1130	26.89	29.93	1.1185	27.98	31.30	1.1240	29.08	32.68
1.1076	25.73	28.51	1.1131	26.91	29.95	1.1186	28.00	31.32	1.1241	29.10	32.71
1.1077	25.75	28.53	1.1132	26.93	29.97	1.1187	28.02	31.35	1.1242	29.12	32.73
1.1078	25.78	28.56	1.1133	26.95	30.00	1.1188	28.04	31.37	1.1243	29.14	32.76
1.1079	25.80	28.58	1.1134	26.97	30.02	1.1189	28.07	31.40	1.1244	29.16	32.78
1.1080	25.82	28.61	1.1135	26.99	30.06	1.1190	28.09	31.43	1.1245	29.18	32.81
1.1081	25.84	28.64	1.1136	27.01	30.08	1.1191	28.11	31.45	1.1246	29.20	32.83
1.1082	25.86	28.66	1.1137	27.03	30.10	1.1192	28.13	31.48	1.1247	29.22	32.86
1.1083	25.89	28.69	1.1138	27.05	30.13	1.1193	28.15	31.51	1.1248	29.24	32.89
1.1084	25.91	28.72	1.1139	27.07	30.15	1.1194	28.17	31.53	1.1249	29.26	32.91
1.1085	25.93	28.75	1.1140	27.09	30.18	1.1195	28.19	31.56	1.1250	29.28	32.94
1.1086	25.96	28.78	1.1141	27.11	30.20	1.1196	28.21	31.59	1.1251	29.30	32.96
1.1087	25.98	28.80	1.1142	27.13	30.22	1.1197	28.23	31.61	1.1252	29.32	32.99
1.1088	26.01	28.83	1.1143	27.15	30.25	1.1198	28.25	31.63	1.1253	29.34	33.02
1.1089	26.03	28.86	1.1144	27.17	30.27	1.1199	28.27	31.65	1.1254	29.36	33.04
1.1090	26.05	28.89	1.1145	27.19	30.31	1.1200	28.28	31.68	1.1255	29.38	33.07
1.1091	26.07	28.92	1.1146	27.21	30.33	1.1201	28.30	31.70	1.1256	29.40	33.09
1.1092	26.09	28.94	1.1147	27.23	30.35	1.1202	28.32	31.73	1.1257	29.42	33.12
1.1093	26.12	28.97	1.1148	27.25	30.37	1.1203	28.34	31.75	1.1258	29.45	33.14
1.1094	26.14	29.00	1.1149	27.27	30.40	1.1204	28.36	31.78	1.1259	29.47	33.17

TABLE IV.—*Extract in beer wort.* ^a

[According to H. Ellion.]

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0001	0.02	0.02	1.0066	1.63	1.64	1.0131	3.22	3.26	1.0196	4.79	4.89
1.0002	0.05	0.05	1.0067	1.66	1.67	1.0132	3.25	3.29	1.0197	4.82	4.91
1.0003	0.07	0.07	1.0068	1.68	1.69	1.0133	3.27	3.31	1.0198	4.84	4.94
1.0004	0.10	0.10	1.0069	1.71	1.72	1.0134	3.29	3.34	1.0199	4.87	4.96
1.0005	0.12	0.12	1.0070	1.73	1.74	1.0135	3.32	3.36	1.0200	4.89	4.99
1.0006	0.15	0.15	1.0071	1.76	1.77	1.0136	3.34	3.39	1.0201	4.91	5.01
1.0007	0.17	0.17	1.0072	1.78	1.79	1.0137	3.37	3.41	1.0202	4.94	5.04
1.0008	0.20	0.20	1.0073	1.80	1.82	1.0138	3.39	3.44	1.0203	4.96	5.06
1.0009	0.22	0.22	1.0074	1.83	1.84	1.0139	3.42	3.46	1.0204	4.99	5.09
1.0010	0.25	0.25	1.0075	1.85	1.87	1.0140	3.44	3.49	1.0205	5.01	5.11
1.0011	0.27	0.27	1.0076	1.88	1.89	1.0141	3.46	3.51	1.0206	5.03	5.14
1.0012	0.30	0.30	1.0077	1.90	1.92	1.0142	3.49	3.54	1.0207	5.06	5.16
1.0013	0.32	0.32	1.0078	1.93	1.94	1.0143	3.51	3.56	1.0208	5.08	5.19
1.0014	0.35	0.35	1.0079	1.95	1.97	1.0144	3.54	3.59	1.0209	5.11	5.21
1.0015	0.37	0.37	1.0080	1.98	1.99	1.0145	3.56	3.61	1.0210	5.13	5.24
1.0016	0.40	0.40	1.0081	2.00	2.02	1.0146	3.59	3.64	1.0211	5.15	5.26
1.0017	0.42	0.42	1.0082	2.03	2.04	1.0147	3.61	3.66	1.0212	5.18	5.29
1.0018	0.45	0.45	1.0083	2.05	2.07	1.0148	3.63	3.69	1.0213	5.20	5.31
1.0019	0.47	0.47	1.0084	2.07	2.09	1.0149	3.66	3.71	1.0214	5.23	5.34
1.0020	0.50	0.50	1.0085	2.10	2.12	1.0150	3.68	3.74	1.0215	5.25	5.36
1.0021	0.52	0.52	1.0086	2.12	2.14	1.0151	3.71	3.76	1.0216	5.27	5.39
1.0022	0.55	0.55	1.0087	2.15	2.17	1.0152	3.73	3.79	1.0217	5.30	5.41
1.0023	0.57	0.57	1.0088	2.17	2.19	1.0153	3.76	3.81	1.0218	5.32	5.44
1.0024	0.60	0.60	1.0089	2.20	2.22	1.0154	3.78	3.84	1.0219	5.35	5.46
1.0025	0.62	0.62	1.0090	2.22	2.24	1.0155	3.80	3.86	1.0220	5.37	5.49
1.0026	0.65	0.65	1.0091	2.25	2.27	1.0156	3.83	3.89	1.0221	5.39	5.51
1.0027	0.67	0.67	1.0092	2.27	2.29	1.0157	3.85	3.91	1.0222	5.42	5.54
1.0028	0.69	0.70	1.0093	2.29	2.32	1.0158	3.88	3.94	1.0223	5.44	5.56
1.0029	0.72	0.72	1.0094	2.32	2.34	1.0159	3.90	3.96	1.0224	5.47	5.59
1.0030	0.74	0.75	1.0095	2.34	2.37	1.0160	3.93	3.99	1.0225	5.49	5.61
1.0031	0.77	0.77	1.0096	2.37	2.39	1.0161	3.95	4.01	1.0226	5.51	5.64
1.0032	0.79	0.80	1.0097	2.39	2.42	1.0162	3.97	4.04	1.0227	5.54	5.66
1.0033	0.82	0.82	1.0098	2.42	2.44	1.0163	4.00	4.06	1.0228	5.56	5.69
1.0034	0.84	0.85	1.0099	2.44	2.47	1.0164	4.02	4.09	1.0229	5.59	5.71
1.0035	0.87	0.87	1.0100	2.47	2.49	1.0165	4.05	4.11	1.0230	5.61	5.74
1.0036	0.89	0.90	1.0101	2.49	2.52	1.0166	4.07	4.14	1.0231	5.63	5.76
1.0037	0.92	0.92	1.0102	2.51	2.54	1.0167	4.09	4.16	1.0232	5.66	5.79
1.0038	0.94	0.95	1.0103	2.54	2.57	1.0168	4.12	4.19	1.0233	5.68	5.81
1.0039	0.97	0.97	1.0104	2.56	2.59	1.0169	4.14	4.21	1.0234	5.70	5.84
1.0040	0.99	1.00	1.0105	2.59	2.62	1.0170	4.17	4.24	1.0235	5.73	5.86
1.0041	1.02	1.02	1.0106	2.61	2.64	1.0171	4.19	4.26	1.0236	5.75	5.89
1.0042	1.04	1.05	1.0107	2.64	2.67	1.0172	4.22	4.29	1.0237	5.78	5.91
1.0043	1.07	1.07	1.0108	2.66	2.69	1.0173	4.24	4.31	1.0238	5.80	5.94
1.0044	1.09	1.10	1.0109	2.69	2.72	1.0174	4.26	4.34	1.0239	5.82	5.96
1.0045	1.12	1.12	1.0110	2.71	2.74	1.0175	4.29	4.36	1.0240	5.85	5.99
1.0046	1.14	1.14	1.0111	2.73	2.77	1.0176	4.31	4.39	1.0241	5.87	6.01
1.0047	1.16	1.17	1.0112	2.76	2.79	1.0177	4.34	4.41	1.0242	5.90	6.04
1.0048	1.19	1.19	1.0113	2.78	2.81	1.0178	4.36	4.44	1.0243	5.92	6.06
1.0049	1.21	1.22	1.0114	2.81	2.84	1.0179	4.38	4.46	1.0244	5.94	6.09
1.0050	1.24	1.24	1.0115	2.83	2.86	1.0180	4.41	4.49	1.0245	5.97	6.11
1.0051	1.26	1.27	1.0116	2.86	2.89	1.0181	4.43	4.51	1.0246	5.99	6.14
1.0052	1.29	1.29	1.0117	2.88	2.91	1.0182	4.46	4.54	1.0247	6.01	6.16
1.0053	1.31	1.32	1.0118	2.91	2.94	1.0183	4.48	4.56	1.0248	6.04	6.19
1.0054	1.34	1.34	1.0119	2.93	2.96	1.0184	4.50	4.59	1.0249	6.06	6.21
1.0055	1.36	1.37	1.0120	2.95	2.99	1.0185	4.53	4.61	1.0250	6.09	6.24
1.0056	1.39	1.39	1.0121	2.98	3.01	1.0186	4.55	4.64	1.0251	6.11	6.26
1.0057	1.41	1.42	1.0122	3.00	3.04	1.0187	4.58	4.66	1.0252	6.13	6.29
1.0058	1.44	1.44	1.0123	3.03	3.06	1.0188	4.60	4.69	1.0253	6.16	6.31
1.0059	1.46	1.47	1.0124	3.05	3.09	1.0189	4.63	4.71	1.0254	6.18	6.34
1.0060	1.48	1.49	1.0125	3.08	3.11	1.0190	4.65	4.74	1.0255	6.21	6.36
1.0061	1.51	1.52	1.0126	3.10	3.14	1.0191	4.67	4.76	1.0256	6.23	6.39
1.0062	1.53	1.54	1.0127	3.12	3.16	1.0192	4.70	4.79	1.0257	6.25	6.41
1.0063	1.57	1.56	1.0128	3.15	3.19	1.0193	4.72	4.81	1.0258	6.28	6.44
1.0064	1.58	1.59	1.0129	3.17	3.21	1.0194	4.75	4.84	1.0259	6.30	6.46
1.0065	1.61	1.62	1.0130	3.20	3.24	1.0195	4.77	4.86	1.0260	6.32	6.49

^a Calculated from results obtained by drying at 97° C.

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0261	6.35	6.51	1.0326	7.89	8.14	1.0391	9.41	9.77	1.0456	10.91	11.41
1.0262	6.37	6.54	1.0327	7.91	8.17	1.0392	9.43	9.80	1.0457	10.93	11.43
1.0263	6.40	6.56	1.0328	7.93	8.19	1.0393	9.45	9.82	1.0458	10.96	11.46
1.0264	6.42	6.59	1.0329	7.96	8.22	1.0394	9.48	9.85	1.0459	10.98	11.48
1.0265	6.44	6.61	1.0330	7.98	8.24	1.0395	9.50	9.87	1.0460	11.00	11.51
1.0266	6.47	6.64	1.0331	8.00	8.27	1.0396	9.52	9.90	1.0461	11.03	11.53
1.0267	6.49	6.66	1.0332	8.03	8.29	1.0397	9.55	9.92	1.0462	11.05	11.56
1.0268	6.51	6.69	1.0333	8.05	8.32	1.0398	9.57	9.95	1.0463	11.07	11.58
1.0269	6.54	6.71	1.0334	8.07	8.34	1.0399	9.59	9.97	1.0464	11.09	11.61
1.0270	6.56	6.74	1.0335	8.10	8.37	1.0400	9.62	10.00	1.0465	11.12	11.63
1.0271	6.59	6.76	1.0336	8.12	8.39	1.0401	9.64	10.03	1.0466	11.14	11.66
1.0272	6.61	6.79	1.0337	8.14	8.42	1.0402	9.66	10.05	1.0467	11.16	11.68
1.0273	6.63	6.81	1.0338	8.17	8.44	1.0403	9.69	10.08	1.0468	11.19	11.71
1.0274	6.66	6.84	1.0339	8.19	8.47	1.0404	9.71	10.10	1.0469	11.21	11.74
1.0275	6.68	6.86	1.0340	8.21	8.49	1.0405	9.73	10.13	1.0470	11.23	11.76
1.0276	6.70	6.89	1.0341	8.24	8.52	1.0406	9.75	10.15	1.0471	11.26	11.79
1.0277	6.73	6.91	1.0342	8.26	8.54	1.0407	9.78	10.18	1.0472	11.28	11.81
1.0278	6.75	6.94	1.0343	8.28	8.57	1.0408	9.80	10.20	1.0473	11.30	11.84
1.0279	6.78	6.96	1.0344	8.31	8.59	1.0409	9.82	10.23	1.0474	11.32	11.86
1.0280	6.80	6.99	1.0345	8.33	8.62	1.0410	9.85	10.25	1.0475	11.35	11.89
1.0281	6.82	7.01	1.0346	8.36	8.64	1.0411	9.87	10.28	1.0476	11.37	11.91
1.0282	6.85	7.04	1.0347	8.38	8.67	1.0412	9.89	10.30	1.0477	11.39	11.94
1.0283	6.87	7.06	1.0348	8.40	8.69	1.0413	9.92	10.33	1.0478	11.42	11.96
1.0284	6.89	7.09	1.0349	8.43	8.72	1.0414	9.94	10.35	1.0479	11.44	11.99
1.0285	6.92	7.11	1.0350	8.45	8.74	1.0415	9.96	10.38	1.0480	11.46	12.01
1.0286	6.94	7.14	1.0351	8.47	8.77	1.0416	9.99	10.40	1.0481	11.48	12.04
1.0287	6.97	7.16	1.0352	8.50	8.79	1.0417	10.01	10.43	1.0482	11.51	12.06
1.0288	6.99	7.19	1.0353	8.52	8.82	1.0418	10.03	10.45	1.0483	11.53	12.09
1.0289	7.01	7.22	1.0354	8.54	8.84	1.0419	10.06	10.48	1.0484	11.55	12.11
1.0290	7.04	7.24	1.0355	8.57	8.87	1.0420	10.08	10.50	1.0485	11.58	12.14
1.0291	7.06	7.27	1.0356	8.59	8.90	1.0421	10.10	10.53	1.0486	11.60	12.16
1.0292	7.08	7.29	1.0357	8.61	8.92	1.0422	10.13	10.55	1.0487	11.62	12.19
1.0293	7.11	7.32	1.0358	8.64	8.95	1.0423	10.15	10.58	1.0488	11.65	12.21
1.0294	7.13	7.34	1.0359	8.66	8.97	1.0424	10.17	10.60	1.0489	11.67	12.24
1.0295	7.15	7.37	1.0360	8.68	9.00	1.0425	10.20	10.63	1.0490	11.69	12.26
1.0296	7.18	7.39	1.0361	8.71	9.02	1.0426	10.22	10.65	1.0491	11.71	12.29
1.0297	7.20	7.42	1.0362	8.73	9.05	1.0427	10.24	10.68	1.0492	11.74	12.31
1.0298	7.23	7.44	1.0363	8.75	9.07	1.0428	10.26	10.70	1.0493	11.76	12.34
1.0299	7.25	7.47	1.0364	8.78	9.10	1.0429	10.29	10.73	1.0494	11.78	12.36
1.0300	7.27	7.49	1.0365	8.80	9.12	1.0430	10.31	10.75	1.0495	11.81	12.39
1.0301	7.30	7.52	1.0366	8.82	9.15	1.0431	10.33	10.78	1.0496	11.83	12.42
1.0302	7.32	7.54	1.0367	8.85	9.17	1.0432	10.36	10.80	1.0497	11.85	12.44
1.0303	7.34	7.57	1.0368	8.87	9.20	1.0433	10.38	10.83	1.0498	11.87	12.47
1.0304	7.37	7.59	1.0369	8.89	9.22	1.0434	10.40	10.85	1.0499	11.90	12.49
1.0305	7.39	7.62	1.0370	8.92	9.25	1.0435	10.43	10.88	1.0500	11.92	12.52
1.0306	7.41	7.64	1.0371	8.94	9.27	1.0436	10.45	10.90	1.0501	11.94	12.54
1.0307	7.44	7.67	1.0372	8.96	9.30	1.0437	10.47	10.93	1.0502	11.97	12.57
1.0308	7.46	7.69	1.0373	8.99	9.32	1.0438	10.50	10.96	1.0503	11.99	12.59
1.0309	7.49	7.72	1.0374	9.01	9.35	1.0439	10.52	10.98	1.0504	12.01	12.62
1.0310	7.51	7.74	1.0375	9.03	9.37	1.0440	10.54	11.01	1.0505	12.03	12.64
1.0311	7.53	7.77	1.0376	9.06	9.40	1.0441	10.56	11.03	1.0506	12.06	12.67
1.0312	7.56	7.79	1.0377	9.08	9.42	1.0442	10.59	11.06	1.0507	12.08	12.69
1.0313	7.58	7.82	1.0378	9.10	9.45	1.0443	10.61	11.08	1.0508	12.10	12.72
1.0314	7.60	7.84	1.0379	9.13	9.47	1.0444	10.63	11.11	1.0509	12.13	12.74
1.0315	7.63	7.87	1.0380	9.15	9.50	1.0445	10.66	11.13	1.0510	12.15	12.77
1.0316	7.65	7.89	1.0381	9.17	9.52	1.0446	10.68	11.16	1.0511	12.17	12.79
1.0317	7.67	7.92	1.0382	9.20	9.55	1.0447	10.70	11.18	1.0512	12.19	12.82
1.0318	7.70	7.94	1.0383	9.22	9.57	1.0448	10.73	11.21	1.0513	12.22	12.84
1.0319	7.72	7.97	1.0384	9.24	9.60	1.0449	10.75	11.23	1.0514	12.24	12.87
1.0320	7.74	7.99	1.0385	9.27	9.62	1.0450	10.77	11.26	1.0515	12.26	12.89
1.0321	7.77	8.02	1.0386	9.29	9.65	1.0451	10.80	11.28	1.0516	12.28	12.92
1.0322	7.79	8.04	1.0387	9.31	9.67	1.0452	10.82	11.31	1.0517	12.31	12.94
1.0323	7.81	8.07	1.0388	9.34	9.70	1.0453	10.84	11.33	1.0518	12.33	12.97
1.0324	7.84	8.09	1.0389	9.36	9.72	1.0454	10.86	11.36	1.0519	12.35	12.99
1.0325	7.86	8.12	1.0390	9.38	9.75	1.0455	10.88	11.38	1.0520	12.38	13.02

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0521	12.40	13.04	1.0586	13.87	14.68	1.0651	15.33	16.33	1.0716	16.77	17.97
1.0522	12.42	13.07	1.0587	13.89	14.71	1.0652	15.35	16.35	1.0717	16.97	18.00
1.0523	12.44	13.10	1.0588	13.92	14.73	1.0653	15.37	16.38	1.0718	16.82	18.02
1.0524	12.47	13.12	1.0589	15.94	14.76	1.0654	15.40	16.40	1.0719	16.84	18.05
1.0525	12.49	13.15	1.0590	13.96	14.79	1.0655	15.42	16.43	1.0720	16.86	18.07
1.0526	12.51	13.17	1.0591	13.98	14.81	1.0656	15.44	16.45	1.0721	16.88	18.10
1.0527	12.54	13.20	1.0592	14.01	14.84	1.0657	15.46	16.48	1.0722	16.90	18.12
1.0528	12.56	13.22	1.0593	14.03	14.86	1.0658	15.48	16.50	1.0723	16.93	18.15
1.0529	12.58	13.25	1.0594	14.05	14.89	1.0659	15.51	16.53	1.0724	16.95	18.17
1.0530	12.60	13.27	1.0595	14.07	14.91	1.0660	15.53	16.55	1.0725	16.97	18.20
1.0531	12.63	13.30	1.0596	14.10	14.94	1.0661	15.55	16.58	1.0726	16.99	18.23
1.0532	12.65	13.32	1.0597	14.12	14.96	1.0662	15.57	16.60	1.0727	17.01	18.25
1.0533	12.67	13.35	1.0598	14.14	14.99	1.0663	15.60	16.63	1.0728	17.04	18.28
1.0534	12.69	13.37	1.0599	14.16	15.01	1.0664	15.62	16.66	1.0729	17.06	18.30
1.0535	12.72	13.40	1.0600	14.19	15.04	1.0665	15.64	16.68	1.0730	17.08	18.33
1.0536	12.74	13.42	1.0601	14.21	15.06	1.0666	15.66	16.71	1.0731	17.10	18.35
1.0537	12.76	13.45	1.0602	14.23	15.09	1.0667	15.69	16.73	1.0732	17.12	18.38
1.0538	12.79	13.47	1.0603	14.25	15.11	1.0668	15.71	16.76	1.0733	17.15	18.40
1.0539	12.81	13.50	1.0604	14.28	15.14	1.0669	15.73	16.78	1.0734	17.17	18.43
1.0540	12.83	13.52	1.0605	14.30	15.16	1.0670	15.75	16.81	1.0735	17.19	18.45
1.0541	12.85	13.55	1.0606	14.32	15.19	1.0671	15.77	16.83	1.0736	17.21	18.48
1.0542	12.88	13.57	1.0607	14.34	15.21	1.0672	15.80	16.86	1.0737	17.23	18.50
1.0543	12.90	13.60	1.0608	14.37	15.24	1.0673	15.82	16.88	1.0738	17.26	18.53
1.0544	12.92	13.62	1.0609	14.39	15.27	1.0674	15.84	16.91	1.0739	17.28	18.55
1.0545	12.94	13.65	1.0610	14.41	15.29	1.0675	15.86	16.93	1.0740	17.30	18.58
1.0546	12.97	13.68	1.0611	14.43	15.32	1.0676	15.89	16.96	1.0741	17.32	18.61
1.0547	12.99	13.70	1.0612	14.46	15.34	1.0677	15.91	16.98	1.0742	17.34	18.63
1.0548	13.01	13.73	1.0613	14.48	15.37	1.0678	15.93	17.01	1.0743	17.37	18.66
1.0549	13.04	13.75	1.0614	14.50	15.39	1.0679	15.95	17.03	1.0744	17.39	18.68
1.0550	13.06	13.78	1.0615	14.52	15.42	1.0680	15.97	17.06	1.0745	17.41	18.71
1.0551	13.08	13.80	1.0616	14.55	15.44	1.0681	16.00	17.09	1.0746	17.43	18.73
1.0552	13.10	13.83	1.0617	14.57	15.47	1.0682	16.02	17.11	1.0747	17.45	18.76
1.0553	13.13	13.85	1.0618	14.59	15.49	1.0683	16.04	17.14	1.0748	17.48	18.78
1.0554	13.15	13.88	1.0619	14.61	15.52	1.0684	16.06	17.16	1.0749	17.50	18.81
1.0555	13.17	13.90	1.0620	14.64	15.54	1.0685	16.09	17.19	1.0750	17.52	18.83
1.0556	13.19	13.93	1.0621	14.66	15.57	1.0686	16.11	17.21	1.0751	17.54	18.86
1.0557	13.22	13.95	1.0622	14.68	15.59	1.0687	16.13	17.24	1.0752	17.56	18.88
1.0558	13.24	13.98	1.0623	14.70	15.62	1.0688	16.15	17.26	1.0753	17.59	18.91
1.0559	13.26	14.00	1.0624	14.73	15.64	1.0689	16.17	17.29	1.0754	17.61	18.93
1.0560	13.28	14.03	1.0625	14.75	15.67	1.0690	16.20	17.31	1.0755	17.63	18.96
1.0561	13.31	14.05	1.0626	14.77	15.69	1.0691	16.22	17.34	1.0756	17.65	18.99
1.0562	13.33	14.08	1.0627	14.79	15.72	1.0692	16.24	17.36	1.0757	17.67	19.01
1.0563	13.35	14.10	1.0628	14.81	15.75	1.0693	16.26	17.39	1.0758	17.69	19.04
1.0564	13.37	14.13	1.0629	14.84	15.77	1.0694	16.28	17.41	1.0759	17.72	19.06
1.0565	13.40	14.15	1.0630	14.86	15.80	1.0695	16.31	17.44	1.0760	17.74	19.09
1.0566	13.42	14.18	1.0631	14.88	15.82	1.0696	16.33	17.47	1.0761	17.76	19.11
1.0567	13.44	14.20	1.0632	14.90	15.85	1.0697	16.35	17.49	1.0762	17.78	19.14
1.0568	13.47	14.23	1.0633	14.93	15.87	1.0698	16.37	17.52	1.0763	17.80	19.16
1.0569	13.49	14.26	1.0634	14.95	15.90	1.0699	16.40	17.54	1.0764	17.83	19.19
1.0570	13.51	14.28	1.0635	14.97	15.92	1.0700	16.42	17.57	1.0765	17.85	19.21
1.0571	13.53	14.31	1.0636	14.99	15.95	1.0701	16.44	17.59	1.0766	17.87	19.24
1.0572	13.56	14.33	1.0637	15.02	15.97	1.0702	16.46	17.62	1.0767	17.89	19.26
1.0573	13.58	14.36	1.0638	15.04	16.00	1.0703	16.48	17.64	1.0768	17.91	19.29
1.0574	13.60	14.38	1.0639	15.06	16.02	1.0704	16.51	17.67	1.0769	17.94	19.32
1.0575	13.62	14.41	1.0640	15.08	16.05	1.0705	16.53	17.69	1.0770	17.96	19.34
1.0576	13.65	14.43	1.0641	15.11	16.07	1.0706	16.55	17.72	1.0771	17.98	19.37
1.0577	13.67	14.46	1.0642	15.13	16.10	1.0707	16.57	17.74	1.0772	18.00	19.39
1.0578	13.69	14.48	1.0643	15.15	16.12	1.0708	16.59	17.77	1.0773	18.02	19.42
1.0579	13.71	14.51	1.0644	15.17	16.15	1.0709	16.62	17.79	1.0774	18.05	19.44
1.0580	13.74	14.53	1.0645	15.19	16.18	1.0710	16.64	17.82	1.0775	18.07	19.47
1.0581	13.76	14.56	1.0646	15.22	16.20	1.0711	16.66	17.85	1.0776	18.09	19.49
1.0582	13.78	14.58	1.0647	15.24	16.23	1.0712	16.68	17.87	1.0777	18.11	19.52
1.0583	13.80	14.61	1.0648	15.26	16.25	1.0713	16.70	17.90	1.0778	18.13	19.54
1.0584	13.83	14.63	1.0649	15.28	16.28	1.0714	16.73	17.92	1.0779	18.15	19.57
1.0585	13.85	14.66	1.0650	15.31	16.30	1.0715	16.75	17.95	1.0780	18.18	19.59

TABLE IV.—*Extract in beer wort*—Continued.

Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.		Specific gravity at 15° C.	Extract.	
	Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.		Per cent by weight.	Grams per 100 cc.
1.0781	18.20	19.62	1.0836	19.39	21.02	1.0891	20.58	22.41	1.0946	21.76	23.81
1.0782	18.22	19.64	1.0837	19.42	21.04	1.0892	20.60	22.44	1.0947	21.78	23.84
1.0783	18.24	19.67	1.0838	19.44	21.07	1.0893	20.62	22.47	1.0948	21.80	23.87
1.0784	18.26	19.70	1.0839	19.46	21.09	1.0894	20.65	22.49	1.0949	21.82	23.89
1.0785	18.29	19.72	1.0840	19.48	21.12	1.0895	20.67	22.52	1.0950	21.84	23.92
1.0786	18.31	19.75	1.0841	19.50	21.14	1.0896	20.69	22.54	1.0951	21.86	23.94
1.0787	18.33	19.77	1.0842	19.52	21.17	1.0897	20.71	22.57	1.0952	21.88	23.97
1.0788	18.35	19.80	1.0843	19.55	21.19	1.0898	20.73	22.59	1.0953	21.91	23.99
1.0789	18.37	19.82	1.0844	19.57	21.22	1.0899	20.75	22.62	1.0954	21.93	24.02
1.0790	18.39	19.85	1.0845	19.59	21.24	1.0900	20.77	22.64	1.0955	21.95	24.04
1.0791	18.42	19.87	1.0846	19.61	21.27	1.0901	20.79	22.67	1.0956	21.97	24.07
1.0792	18.44	19.90	1.0847	19.63	21.30	1.0902	20.82	22.69	1.0957	21.99	24.09
1.0793	18.46	19.92	1.0848	19.65	21.32	1.0903	20.84	22.72	1.0958	22.01	24.12
1.0794	18.48	19.95	1.0849	19.68	21.35	1.0904	20.86	22.75	1.0959	22.03	24.15
1.0795	18.50	19.97	1.0850	19.70	21.37	1.0905	20.88	22.77	1.0960	22.05	24.17
1.0796	18.53	20.00	1.0851	19.72	21.40	1.0906	20.90	22.80	1.0961	22.08	24.20
1.0797	18.55	20.03	1.0852	19.74	21.42	1.0907	20.92	22.82	1.0962	22.10	24.22
1.0798	18.57	20.05	1.0853	19.76	21.45	1.0908	20.94	22.85	1.0963	22.12	24.25
1.0799	18.59	20.08	1.0854	19.78	21.47	1.0909	20.97	22.87	1.0964	22.14	24.27
1.0800	18.61	20.10	1.0855	19.81	21.50	1.0910	20.99	22.90	1.0965	22.16	24.30
1.0801	18.63	20.13	1.0856	19.83	21.52	1.0911	21.01	22.92	1.0966	22.18	24.32
1.0802	18.66	20.15	1.0857	19.85	21.55	1.0912	21.03	22.95	1.0967	22.20	24.35
1.0803	18.68	20.18	1.0858	19.87	21.58	1.0913	21.05	22.97	1.0968	22.22	24.38
1.0804	18.70	20.20	1.0859	19.89	21.60	1.0914	21.07	23.00	1.0969	22.25	24.40
1.0805	18.72	20.23	1.0860	19.91	21.63	1.0915	21.09	23.02	1.0970	22.27	24.43
1.0806	18.74	20.25	1.0861	19.93	21.65	1.0916	21.12	23.05	1.0971	22.29	24.45
1.0807	18.77	20.28	1.0862	19.96	21.68	1.0917	21.14	23.08	1.0972	22.31	24.48
1.0808	18.79	20.30	1.0863	19.98	21.70	1.0918	21.16	23.10	1.0973	22.33	24.50
1.0809	18.81	20.33	1.0864	20.00	21.73	1.0919	21.18	23.13	1.0974	22.35	24.53
1.0810	18.83	20.36	1.0865	20.02	21.75	1.0920	21.20	23.15	1.0975	22.37	24.55
1.0811	18.85	20.38	1.0866	20.04	21.78	1.0921	21.22	23.18	1.0976	22.39	24.58
1.0812	18.87	20.41	1.0867	20.06	21.80	1.0922	21.24	23.20	1.0977	22.41	24.60
1.0813	18.90	20.43	1.0868	20.09	21.83	1.0923	21.27	23.23	1.0978	22.44	24.63
1.0814	18.92	20.46	1.0869	20.11	21.85	1.0924	21.29	23.25	1.0979	22.46	24.66
1.0815	18.94	20.48	1.0870	20.13	21.88	1.0925	21.31	33.28	1.0980	22.48	24.68
1.0816	18.96	20.51	1.0871	20.15	21.91	1.0926	21.33	23.31	1.0981	22.50	24.71
1.0817	18.98	20.53	1.0872	20.17	21.93	1.0927	21.35	23.33	1.0982	22.52	24.73
1.0818	19.00	20.56	1.0873	20.19	21.96	1.0928	21.37	23.36	1.0983	22.54	24.76
1.0819	19.03	20.58	1.0874	25.21	21.98	1.0929	21.39	23.38	1.0984	22.56	25.78
1.0820	19.05	20.61	1.0875	20.24	22.01	1.0930	21.42	23.41	1.0985	22.58	24.81
1.0821	19.07	20.63	1.0876	20.26	22.03	1.0931	21.44	23.43	1.0986	22.61	24.83
1.0822	19.09	20.66	1.0877	20.28	22.06	1.0932	21.46	23.46	1.0987	22.63	24.86
1.0823	19.11	20.69	1.0878	20.30	22.08	1.0933	21.48	23.48	1.0988	22.65	24.89
1.0824	19.13	20.71	1.0879	20.32	22.11	1.0934	21.50	23.51	1.0989	22.67	24.91
1.0825	19.16	20.74	1.0880	20.34	22.13	1.0935	21.52	23.53	1.0990	22.69	24.94
1.0826	19.18	20.76	1.0881	20.37	22.16	1.0936	21.54	23.56	1.0991	22.71	24.96
1.0827	19.20	20.79	1.0882	10.39	22.19	1.0937	21.56	23.59	1.0992	22.73	24.99
1.0828	19.22	20.81	1.0883	20.41	22.21	1.0938	21.59	23.61			
1.0829	19.24	20.84	1.0884	20.43	22.24	1.0939	21.61	23.64			
1.0830	19.26	20.86	1.0885	20.45	22.26	1.0940	21.63	23.66			
1.0831	19.29	20.89	1.0886	20.47	22.29	1.0941	21.65	23.69			
1.0832	19.31	20.91	1.0887	20.49	22.31	1.0942	21.67	23.71			
1.0833	19.33	20.94	1.0888	20.52	22.34	1.0943	21.69	23.74			
1.0834	19.35	20.96	1.0889	20.54	22.36	1.0944	21.71	23.76			
1.0835	19.37	20.99	1.0890	20.56	22.39	1.0945	21.73	23.79			

TABLE V.—*Extract in wine.*

[According to Windisch.]

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0000	0.00	1.0065	1.68	1.0130	3.36	1.0195	5.04	1.0260	6.72	1.0325	8.40
1.0001	0.03	1.0066	1.70	1.0131	3.38	1.0196	5.06	1.0261	6.75	1.0326	8.43
1.0002	0.05	1.0067	1.73	1.0132	3.41	1.0197	5.09	1.0262	6.77	1.0327	8.46
1.0003	0.08	1.0068	1.76	1.0133	3.43	1.0198	5.11	1.0263	6.80	1.0328	8.48
1.0004	0.10	1.0069	1.78	1.0134	3.46	1.0199	5.14	1.0264	6.82	1.0329	8.51
1.0005	0.13	1.0070	1.81	1.0135	3.49	1.0200	5.17	1.0265	6.85	1.0330	8.53
1.0006	0.15	1.0071	1.83	1.0136	3.51	1.0201	5.19	1.0266	6.88	1.0331	8.56
1.0007	0.18	1.0072	1.86	1.0137	3.54	1.0202	5.22	1.0267	6.90	1.0332	8.59
1.0008	0.20	1.0073	1.88	1.0138	3.56	1.0203	5.25	1.0268	6.93	1.0333	8.61
1.0009	0.23	1.0074	1.91	1.0139	3.59	1.0204	5.27	1.0269	6.95	1.0334	8.64
1.0010	0.26	1.0075	1.94	1.0140	3.62	1.0205	5.30	1.0270	6.98	1.0335	8.66
1.0011	0.28	1.0076	1.96	1.0141	3.64	1.0206	5.32	1.0271	7.01	1.0336	8.69
1.0012	0.31	1.0077	1.99	1.0142	3.67	1.0207	5.35	1.0272	7.03	1.0337	8.72
1.0013	0.34	1.0078	2.01	1.0143	3.69	1.0208	5.38	1.0273	7.06	1.0338	8.74
1.0014	0.36	1.0079	2.04	1.0144	3.72	1.0209	5.40	1.0274	7.08	1.0339	8.77
1.0015	0.39	1.0080	2.07	1.0145	3.75	1.0210	5.43	1.0275	7.11	1.0340	8.79
1.0016	0.41	1.0081	2.09	1.0146	3.77	1.0211	5.45	1.0276	7.13	1.0341	8.82
1.0017	0.44	1.0082	2.12	1.0147	3.80	1.0212	5.48	1.0277	7.16	1.0342	8.85
1.0018	0.46	1.0083	2.14	1.0148	3.82	1.0213	5.51	1.0278	7.19	1.0343	8.87
1.0019	0.49	1.0084	2.17	1.0149	3.85	1.0214	5.53	1.0279	7.21	1.0344	8.90
1.0020	0.52	1.0085	2.19	1.0150	3.87	1.0215	5.56	1.0280	7.24	1.0345	8.92
1.0021	0.54	1.0086	2.22	1.0151	3.90	1.0216	5.58	1.0281	7.26	1.0346	8.95
1.0022	0.57	1.0087	2.25	1.0152	3.93	1.0217	5.61	1.0282	7.29	1.0347	8.97
1.0023	0.59	1.0088	2.27	1.0153	3.95	1.0218	5.64	1.0283	7.32	1.0348	9.00
1.0024	0.62	1.0089	2.30	1.0154	3.98	1.0219	5.66	1.0284	7.34	1.0349	9.03
1.0025	0.64	1.0090	2.32	1.0155	4.00	1.0220	5.69	1.0285	7.37	1.0350	9.05
1.0026	0.67	1.0091	2.35	1.0156	4.03	1.0221	5.71	1.0286	7.39	1.0351	9.08
1.0027	0.69	1.0092	2.38	1.0157	4.06	1.0222	5.74	1.0287	7.42	1.0352	9.10
1.0028	0.72	1.0093	2.40	1.0158	4.08	1.0223	5.77	1.0288	7.45	1.0353	9.13
1.0029	0.75	1.0094	2.43	1.0159	4.11	1.0224	5.79	1.0289	7.47	1.0354	9.16
1.0030	0.77	1.0095	2.45	1.0160	4.13	1.0225	5.82	1.0290	7.50	1.0355	9.18
1.0031	0.80	1.0096	2.48	1.0161	4.16	1.0226	5.84	1.0291	7.52	1.0356	9.21
1.0032	0.82	1.0097	2.50	1.0162	4.19	1.0227	5.87	1.0292	7.55	1.0357	9.23
1.0033	0.85	1.0098	2.53	1.0163	4.21	1.0228	5.89	1.0293	7.58	1.0358	9.26
1.0034	0.87	1.0099	2.56	1.0164	4.24	1.0229	5.92	1.0294	7.60	1.0359	9.29
1.0035	0.90	1.0100	2.58	1.0165	4.26	1.0230	5.94	1.0295	7.63	1.0360	9.31
1.0036	0.93	1.0101	2.61	1.0166	4.29	1.0231	5.97	1.0296	7.65	1.0361	9.34
1.0037	0.95	1.0102	2.63	1.0167	4.31	1.0232	6.00	1.0297	7.68	1.0362	9.36
1.0038	0.98	1.0103	2.66	1.0168	4.34	1.0233	6.02	1.0298	7.70	1.0363	9.39
1.0039	1.00	1.0104	2.69	1.0169	4.37	1.0234	6.05	1.0299	7.73	1.0364	9.42
1.0040	1.03	1.0105	2.71	1.0170	4.39	1.0235	6.07	1.0300	7.76	1.0365	9.44
1.0041	1.05	1.0106	2.74	1.0171	4.42	1.0236	6.10	1.0301	7.78	1.0366	9.47
1.0042	1.08	1.0107	2.76	1.0172	4.44	1.0237	6.12	1.0302	7.81	1.0367	9.49
1.0043	1.11	1.0108	2.79	1.0173	4.47	1.0238	6.15	1.0303	7.83	1.0368	9.52
1.0044	1.13	1.0109	2.82	1.0174	4.50	1.0239	6.18	1.0304	7.86	1.0369	9.55
1.0045	1.16	1.0110	2.84	1.0175	4.52	1.0240	6.20	1.0305	7.89	1.0370	9.57
1.0046	1.18	1.0111	2.87	1.0176	4.55	1.0241	6.23	1.0306	7.91	1.0371	9.60
1.0047	1.21	1.0112	2.89	1.0177	4.57	1.0242	6.25	1.0307	7.94	1.0372	9.62
1.0048	1.24	1.0113	2.92	1.0178	4.60	1.0243	6.28	1.0308	7.97	1.0373	9.65
1.0049	1.26	1.0114	2.94	1.0179	4.63	1.0244	6.31	1.0309	7.99	1.0374	9.68
1.0050	1.29	1.0115	2.97	1.0180	4.65	1.0245	6.33	1.0310	8.02	1.0375	9.70
1.0051	1.32	1.0116	3.00	1.0181	4.68	1.0246	6.36	1.0311	8.04	1.0376	9.73
1.0052	1.34	1.0117	3.02	1.0182	4.70	1.0247	6.38	1.0312	8.07	1.0377	9.75
1.0053	1.37	1.0118	3.05	1.0183	4.73	1.0248	6.41	1.0313	8.09	1.0378	9.78
1.0054	1.39	1.0119	3.07	1.0184	4.75	1.0249	6.44	1.0314	8.12	1.0379	9.80
1.0055	1.42	1.0120	3.10	1.0185	4.78	1.0250	6.46	1.0315	8.14	1.0380	9.83
1.0056	1.45	1.0121	3.12	1.0186	4.81	1.0251	6.49	1.0316	8.17	1.0381	9.86
1.0057	1.47	1.0122	3.15	1.0187	4.83	1.0252	6.51	1.0317	8.20	1.0382	9.88
1.0058	1.50	1.0123	3.18	1.0188	4.86	1.0253	6.54	1.0318	8.22	1.0383	9.91
1.0059	1.52	1.0124	3.20	1.0189	4.88	1.0254	6.56	1.0319	8.25	1.0384	9.93
1.0060	1.55	1.0125	3.23	1.0190	4.91	1.0255	6.59	1.0320	8.27	1.0385	9.96
1.0061	1.57	1.0126	3.26	1.0191	4.94	1.0256	6.62	1.0321	8.30	1.0386	9.99
1.0062	1.60	1.0127	3.28	1.0192	4.96	1.0257	6.64	1.0322	8.33	1.0387	10.01
1.0063	1.63	1.0128	3.31	1.0193	4.99	1.0258	6.67	1.0323	8.35	1.0388	10.04
1.0064	1.65	1.0129	3.33	1.0194	5.01	1.0259	6.70	1.0324	8.38	1.0389	10.06

TABLE V.—*Extract in wine*—Continued.

Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.	Specific gravity.	Ex-tract.
1.0390	10.09	1.0455	11.78	1.0520	13.47	1.0585	15.16	1.0650	16.86	1.0715	18.56
1.0391	10.11	1.0456	11.81	1.0521	13.49	1.0586	15.19	1.0651	16.88	1.0716	18.58
1.0392	10.14	1.0457	11.83	1.0522	13.52	1.0587	15.22	1.0652	16.91	1.0717	18.61
1.0393	10.17	1.0458	11.86	1.0523	13.55	1.0588	15.24	1.0653	16.94	1.0718	18.63
1.0394	10.19	1.0459	11.88	1.0524	13.57	1.0589	15.27	1.0654	16.96	1.0719	18.66
1.0395	10.22	1.0460	11.91	1.0525	13.60	1.0590	15.29	1.0655	16.99	1.0720	18.69
1.0396	10.25	1.0461	11.94	1.0526	13.62	1.0591	15.32	1.0656	17.01	1.0721	18.71
1.0397	10.27	1.0462	11.96	1.0527	13.65	1.0592	15.35	1.0657	17.04	1.0722	18.74
1.0398	10.30	1.0463	11.99	1.0528	13.68	1.0593	15.37	1.0658	17.07	1.0723	18.76
1.0399	10.32	1.0464	12.01	1.0529	13.70	1.0594	15.40	1.0659	17.09	1.0724	18.79
1.0400	10.35	1.0465	12.04	1.0530	13.73	1.0595	15.42	1.0660	17.12	1.0725	18.82
1.0401	10.37	1.0466	12.06	1.0531	13.75	1.0596	15.45	1.0661	17.14	1.0726	18.84
1.0402	10.40	1.0467	12.09	1.0532	13.78	1.0597	15.48	1.0662	17.17	1.0727	18.87
1.0403	10.43	1.0468	12.12	1.0533	13.81	1.0598	15.50	1.0663	17.20	1.0728	18.90
1.0404	10.45	1.0469	12.14	1.0534	13.83	1.0599	15.53	1.0664	17.22	1.0729	18.92
1.0405	10.48	1.0470	12.17	1.0535	13.86	1.0600	15.55	1.0665	17.25	1.0730	18.95
1.0406	10.51	1.0471	12.19	1.0536	13.89	1.0601	15.58	1.0666	17.27	1.0731	18.97
1.0407	10.53	1.0472	12.22	1.0537	13.91	1.0602	15.61	1.0667	17.30	1.0732	19.00
1.0408	10.56	1.0473	12.25	1.0538	13.94	1.0603	15.63	1.0668	17.33	1.0733	19.03
1.0409	10.58	1.0474	12.27	1.0539	13.96	1.0604	15.66	1.0669	17.35	1.0734	19.05
1.0410	10.61	1.0475	12.30	1.0540	13.99	1.0605	15.68	1.0670	17.38	1.0735	19.08
1.0411	10.63	1.0476	12.32	1.0541	14.01	1.0606	15.71	1.0671	17.41	1.0736	19.10
1.0412	10.66	1.0477	12.35	1.0542	14.04	1.0607	15.74	1.0672	17.43	1.0737	19.13
1.0413	10.69	1.0478	12.38	1.0543	14.07	1.0608	15.76	1.0673	17.46	1.0738	19.16
1.0414	10.71	1.0479	12.40	1.0544	14.09	1.0609	15.79	1.0674	17.48	1.0739	19.18
1.0415	10.74	1.0480	12.43	1.0545	14.12	1.0610	15.81	1.0675	17.51	1.0740	19.21
1.0416	10.76	1.0481	12.45	1.0546	14.14	1.0611	15.84	1.0676	17.54	1.0741	19.23
1.0417	10.79	1.0482	12.48	1.0547	14.17	1.0612	15.87	1.0677	17.56	1.0742	19.26
1.0418	10.82	1.0483	12.51	1.0548	14.20	1.0613	15.89	1.0678	17.59	1.0743	19.29
1.0419	10.84	1.0484	12.53	1.0549	14.22	1.0614	15.92	1.0679	17.62	1.0744	19.31
1.0420	10.87	1.0485	12.56	1.0550	14.25	1.0615	15.94	1.0680	17.64	1.0745	19.34
1.0421	10.90	1.0486	12.58	1.0551	14.28	1.0616	15.97	1.0681	17.67	1.0746	19.37
1.0422	10.92	1.0487	12.61	1.0552	14.30	1.0617	16.00	1.0682	17.69	1.0747	19.39
1.0423	10.95	1.0488	12.64	1.0553	14.33	1.0618	16.02	1.0683	17.72	1.0748	19.42
1.0424	10.97	1.0489	12.66	1.0554	14.35	1.0619	16.05	1.0684	17.75	1.0749	19.44
1.0425	11.00	1.0490	12.69	1.0555	14.38	1.0620	16.07	1.0685	17.77	1.0750	19.47
1.0426	11.03	1.0491	12.71	1.0556	14.41	1.0621	16.10	1.0686	17.80	1.0751	19.50
1.0427	11.05	1.0492	12.74	1.0557	14.43	1.0622	16.13	1.0687	17.83	1.0752	19.52
1.0428	11.08	1.0493	12.77	1.0558	14.46	1.0623	16.15	1.0688	17.85	1.0753	19.55
1.0429	11.10	1.0494	12.79	1.0559	14.48	1.0624	16.18	1.0689	17.88	1.0754	19.58
1.0430	11.13	1.0495	12.82	1.0560	14.51	1.0625	16.21	1.0690	17.90	1.0755	19.60
1.0431	11.15	1.0496	12.84	1.0561	14.54	1.0626	16.23	1.0691	17.93	1.0756	19.63
1.0432	11.18	1.0497	12.87	1.0562	14.56	1.0627	16.26	1.0692	17.95	1.0757	19.65
1.0433	11.21	1.0498	12.90	1.0563	14.59	1.0628	16.28	1.0693	17.98	1.0758	19.68
1.0434	11.23	1.0499	12.92	1.0564	14.61	1.0629	16.31	1.0694	18.01	1.0759	19.71
1.0435	11.26	1.0500	12.95	1.0565	14.64	1.0630	16.33	1.0695	18.03	1.0760	19.73
1.0436	11.28	1.0501	12.97	1.0566	14.67	1.0631	16.36	1.0696	18.06	1.0761	19.76
1.0437	11.31	1.0502	13.00	1.0567	14.69	1.0632	16.39	1.0697	18.08	1.0762	19.79
1.0438	11.34	1.0503	13.03	1.0568	14.72	1.0633	16.41	1.0698	18.11	1.0763	19.81
1.0439	11.36	1.0504	13.05	1.0569	14.74	1.0634	16.44	1.0699	18.14	1.0764	19.84
1.0440	11.39	1.0505	13.08	1.0570	14.77	1.0635	16.47	1.0700	18.16	1.0765	19.86
1.0441	11.42	1.0506	13.10	1.0571	14.80	1.0636	16.49	1.0701	18.19	1.0766	19.89
1.0442	11.44	1.0507	13.13	1.0572	14.82	1.0637	16.52	1.0702	18.22	1.0767	19.92
1.0443	11.47	1.0508	13.16	1.0573	14.85	1.0638	16.54	1.0703	18.24	1.0768	19.94
1.0444	11.49	1.0509	13.18	1.0574	14.87	1.0639	16.57	1.0704	18.27	1.0769	19.97
1.0445	11.52	1.0510	13.21	1.0575	14.90	1.0640	16.60	1.0705	18.30	1.0770	20.00
1.0446	11.55	1.0511	13.23	1.0576	14.93	1.0641	16.62	1.0706	18.32	1.0771	20.02
1.0447	11.57	1.0512	13.26	1.0577	14.95	1.0642	16.65	1.0707	18.35	1.0772	20.05
1.0448	11.60	1.0513	13.29	1.0578	14.98	1.0643	16.68	1.0708	18.37	1.0773	20.07
1.0449	11.62	1.0514	13.31	1.0579	15.00	1.0644	16.70	1.0709	18.40	1.0774	20.10
1.0450	11.65	1.0515	13.34	1.0580	15.03	1.0645	16.73	1.0710	18.43	1.0775	20.12
1.0451	11.68	1.0516	13.36	1.0581	15.06	1.0646	16.75	1.0711	18.45	1.0776	20.15
1.0452	11.70	1.0517	13.39	1.0582	15.08	1.0647	16.78	1.0712	18.48	1.0777	20.18
1.0453	11.73	1.0518	13.42	1.0583	15.11	1.0648	16.80	1.0713	18.50	1.0778	20.20
1.0454	11.75	1.0519	13.44	1.0584	15.14	1.0649	16.83	1.0714	18.53	1.0779	20.23

TABLE V.—*Extract in wine*—Continued.

Spec- ific gravity.	Ex- tract.	Spe- cific gravity.	Ex- tract.	Spe- cific gravity.	Ex- tract.	Spe- cific gravity.	Ex- tract.	Spe- cific gravity.	Ex- tract.	Spe- cific gravity.	Ex- tract.
1.0780	20.26	1.0845	21.96	1.0910	23.67	1.0975	25.38	1.1040	27.09	1.1105	28.81
1.0781	20.28	1.0846	21.99	1.0911	23.70	1.0976	25.41	1.1041	27.12	1.1106	28.83
1.0782	20.31	1.0847	22.02	1.0912	23.72	1.0977	25.43	1.1042	27.15	1.1107	28.86
1.0783	20.34	1.0848	22.04	1.0913	23.75	1.0978	25.46	1.1043	27.17	1.1108	28.88
1.0784	20.36	1.0849	22.07	1.0914	23.77	1.0979	25.49	1.1044	27.20	1.1109	28.91
1.0785	20.39	1.0850	22.09	1.0915	23.80	1.0980	25.51	1.1045	27.22	1.1110	28.94
1.0786	20.41	1.0851	22.12	1.0916	23.83	1.0981	25.54	1.1046	27.25	1.1111	28.96
1.0787	20.44	1.0852	22.15	1.0917	23.85	1.0982	25.56	1.1047	27.27	1.1112	28.99
1.0788	20.47	1.0853	22.17	1.0918	23.88	1.0983	25.59	1.1048	27.30	1.1113	29.02
1.0789	20.49	1.0854	22.20	1.0919	23.91	1.0984	25.62	1.1049	27.33	1.1114	29.04
1.0790	20.52	1.0855	22.22	1.0920	23.93	1.0985	25.64	1.1050	27.35	1.1115	29.07
1.0791	20.55	1.0856	22.25	1.0921	23.96	1.0986	25.67	1.1051	27.38	1.1116	29.09
1.0792	20.57	1.0857	22.28	1.0922	23.99	1.0987	25.70	1.1052	27.41	1.1117	29.12
1.0793	20.60	1.0858	22.30	1.0923	24.01	1.0988	25.72	1.1053	27.43	1.1118	29.15
1.0794	20.62	1.0859	22.33	1.0924	24.04	1.0989	25.75	1.1054	27.46	1.1119	29.17
1.0795	20.65	1.0860	22.36	1.0925	24.07	1.0990	25.78	1.1055	27.49	1.1120	29.20
1.0796	20.68	1.0861	22.38	1.0926	24.09	1.0991	25.80	1.1056	27.51	1.1121	29.23
1.0797	20.70	1.0862	22.41	1.0927	24.12	1.0992	25.83	1.1057	27.54	1.1122	29.25
1.0798	20.73	1.0863	22.43	1.0928	24.14	1.0993	25.85	1.1058	27.57	1.1123	29.28
1.0799	20.75	1.0864	22.46	1.0929	24.17	1.0994	25.88	1.1059	27.59	1.1124	29.31
1.0800	20.78	1.0865	22.49	1.0930	24.20	1.0995	25.91	1.1060	27.62	1.1125	29.33
1.0801	20.81	1.0866	22.51	1.0931	24.22	1.0996	25.93	1.1061	27.65	1.1126	29.36
1.0802	20.83	1.0867	22.54	1.0932	24.25	1.0997	25.96	1.1062	27.67	1.1127	29.39
1.0803	20.86	1.0868	22.57	1.0933	24.27	1.0998	25.99	1.1063	27.70	1.1128	29.41
1.0804	20.89	1.0869	22.59	1.0934	24.30	1.0999	26.01	1.1064	27.72	1.1129	29.44
1.0805	20.91	1.0870	22.62	1.0935	24.33	1.1000	26.04	1.1065	27.75	1.1130	29.47
1.0806	20.94	1.0871	22.65	1.0936	24.35	1.1001	26.06	1.1066	27.78	1.1131	29.49
1.0807	20.96	1.0872	22.67	1.0937	24.38	1.1002	26.09	1.1067	27.80	1.1132	29.52
1.0808	20.99	1.0873	22.70	1.0938	24.41	1.1003	26.12	1.1068	27.83	1.1133	29.54
1.0809	21.02	1.0874	22.72	1.0939	24.43	1.1004	26.14	1.1069	27.86	1.1134	29.57
1.0810	21.04	1.0875	22.75	1.0940	24.46	1.1005	26.17	1.1070	27.88	1.1135	29.60
1.0811	21.07	1.0876	22.78	1.0941	24.49	1.1006	26.20	1.1071	27.96	1.1136	29.62
1.0812	21.10	1.0877	22.80	1.0942	24.51	1.1007	26.22	1.1072	27.93	1.1137	29.65
1.0813	21.12	1.0878	22.83	1.0943	24.54	1.1008	26.25	1.1073	27.96	1.1138	29.68
1.0814	21.15	1.0879	22.86	1.0944	24.57	1.1009	26.27	1.1074	27.99	1.1139	29.70
1.0815	21.17	1.0880	22.88	1.0945	24.59	1.1010	26.30	1.1075	28.01	1.1140	29.73
1.0816	21.20	1.0881	22.91	1.0946	24.62	1.1011	26.33	1.1076	28.04	1.1141	29.76
1.0817	21.23	1.0882	22.93	1.0947	24.64	1.1012	26.35	1.1077	28.07	1.1142	29.78
1.0818	21.25	1.0883	22.96	1.0948	24.67	1.1013	26.38	1.1078	28.09	1.1143	29.81
1.0819	21.28	1.0884	22.99	1.0949	24.70	1.1014	26.41	1.1079	28.12	1.1144	29.83
1.0820	21.31	1.0885	23.01	1.0950	24.72	1.1015	26.43	1.1080	28.15	1.1145	29.86
1.0821	21.33	1.0886	23.04	1.0951	24.75	1.1016	26.46	1.1081	28.17	1.1146	29.89
1.0822	21.36	1.0887	23.07	1.0952	24.78	1.1017	26.49	1.1082	28.20	1.1147	29.91
1.0823	21.38	1.0888	23.09	1.0953	24.80	1.1018	26.51	1.1083	28.22	1.1148	29.94
1.0824	21.41	1.0889	23.12	1.0954	24.83	1.1019	26.54	1.1084	28.25	1.1149	29.96
1.0825	21.44	1.0890	23.14	1.0955	24.85	1.1020	26.56	1.1085	28.28	1.1150	29.99
1.0826	21.46	1.0891	23.17	1.0956	24.88	1.1021	26.59	1.1086	28.20	1.1151	30.02
1.0827	21.49	1.0892	23.20	1.0957	24.91	1.1022	26.62	1.1087	28.33	1.1152	30.04
1.0828	21.52	1.0893	23.22	1.0958	24.93	1.1023	26.64	1.1088	28.36	1.1153	30.07
1.0829	21.54	1.0894	23.25	1.0959	24.96	1.1024	26.67	1.1089	28.38	1.1154	30.10
1.0830	21.57	1.0895	23.28	1.0960	24.99	1.1025	26.70	1.1090	28.41	1.1155	30.13
1.0831	21.59	1.0896	23.30	1.0961	25.01	1.1026	26.72	1.1091	28.43	1.1156	30.15
1.0832	21.62	1.0897	23.33	1.0962	25.04	1.1027	26.75	1.1092	28.46	1.1157	30.18
1.0833	21.65	1.0898	23.35	1.0963	25.07	1.1028	26.78	1.1093	28.49	1.1158	30.21
1.0834	21.67	1.0899	23.38	1.0964	25.09	1.1029	26.80	1.1094	28.51	1.1159	30.23
1.0835	21.70	1.0900	23.41	1.0965	25.12	1.1030	26.83	1.1095	28.54		
1.0836	21.73	1.0901	23.43	1.0966	25.14	1.1031	26.85	1.1096	28.57		
1.0837	21.75	1.0902	23.46	1.0967	25.17	1.1032	26.88	1.1097	28.59		
1.0838	21.78	1.0903	23.49	1.0968	25.20	1.1033	26.91	1.1098	28.62		
1.0839	21.80	1.0904	23.51	1.0969	25.22	1.1034	26.93	1.1099	28.65		
1.0840	21.83	1.0905	23.54	1.0970	25.25	1.1035	26.96	1.1100	28.67		
1.0841	21.86	1.0906	23.57	1.0971	25.28	1.1036	26.99	1.1101	28.70		
1.0842	21.88	1.0907	23.59	1.0972	25.30	1.1037	27.01	1.1102	28.73		
1.0843	21.91	1.0908	23.62	1.0973	25.33	1.1038	27.04	1.1103	28.75		
1.0844	21.94	1.0909	23.65	1.0974	25.36	1.1039	27.07	1.1104	28.78		

TABLE VI.—*Relation of brix, specific gravity, and Baumé.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
0.1	1.0003	0.06	6.6	1.0261	3.7	13.1	1.0531	7.3	19.6	1.0815	10.85
0.2	1.0007	0.11	6.7	1.0265	3.7	13.2	1.0536	7.3	19.7	1.0819	10.9
0.3	1.0011	0.17	6.8	1.0269	3.8	13.3	1.0540	7.4	19.8	1.0824	11.0
0.4	1.0015	0.22	6.9	1.0273	3.8	13.4	1.0544	7.4	19.9	1.0828	11.0
0.5	1.0019	0.28	7.0	1.0277	3.9	13.5	1.0548	7.5	20.0	1.0832	11.1
0.6	1.0023	0.33	7.1	1.0281	3.9	13.6	1.0553	7.5	20.1	1.0837	11.1
0.7	1.0027	0.39	7.2	1.0286	4.0	13.7	1.0557	7.6	20.2	1.0841	11.2
0.8	1.0031	0.44	7.3	1.0290	4.1	13.8	1.0561	7.65	20.3	1.0846	11.2
0.9	1.0034	0.5	7.4	1.0294	4.1	13.9	1.0566	7.7	20.4	1.0850	11.3
1.0	1.0038	0.55	7.5	1.0298	4.2	14.0	1.0570	7.8	20.5	1.0855	11.3
1.1	1.0042	0.6	7.6	1.0302	4.2	14.1	1.0574	7.8	20.6	1.0859	11.4
1.2	1.0046	0.7	7.7	1.0306	4.3	14.2	1.0578	7.9	20.7	1.0864	11.45
1.3	1.0050	0.7	7.8	1.0310	4.3	14.3	1.0583	7.9	20.8	1.0868	11.5
1.4	1.0054	0.8	7.9	1.0314	4.4	14.4	1.0587	8.0	20.9	1.0873	11.6
1.5	1.0058	0.8	8.0	1.0318	4.4	14.5	1.0591	8.0	21.0	1.0877	11.6
1.6	1.0062	0.9	8.1	1.0322	4.5	14.6	1.0596	8.1	21.1	1.0882	11.7
1.7	1.0066	0.9	8.2	1.0327	4.55	14.7	1.0600	8.15	21.2	1.0886	11.7
1.8	1.0070	1.0	8.3	1.0331	4.6	14.8	1.0604	8.2	21.3	1.0891	11.8
1.9	1.0074	1.05	8.4	1.0335	4.7	14.9	1.0609	8.3	21.4	1.0895	11.8
2.0	1.0077	1.1	8.5	1.0339	4.7	15.0	1.0613	8.3	21.5	1.0900	11.9
2.1	1.0081	1.2	8.6	1.0343	4.8	15.1	1.0617	8.4	21.6	1.0904	11.95
2.2	1.0085	1.2	8.7	1.0347	4.8	15.2	1.0621	8.4	21.7	1.0909	12.0
2.3	1.0089	1.3	8.8	1.0351	4.9	15.3	1.0626	8.5	21.8	1.0914	12.05
2.4	1.0093	1.3	8.9	1.0355	4.9	15.4	1.0630	8.5	21.9	1.0918	12.1
2.5	1.0097	1.4	9.0	1.0359	5.0	15.5	1.0634	8.6	22.0	1.0923	12.2
2.6	1.0101	1.4	9.1	1.0364	5.05	15.6	1.0639	8.65	22.1	1.0927	12.2
2.7	1.0105	1.5	9.2	1.0368	5.1	15.7	1.0643	8.7	22.2	1.0932	12.3
2.8	1.0109	1.55	9.3	1.0372	5.2	15.8	1.0647	8.8	22.3	1.0936	12.3
2.9	1.0113	1.6	9.4	1.0376	5.2	15.9	1.0652	8.8	22.4	1.0941	12.4
3.0	1.0117	1.7	9.5	1.0380	5.3	16.0	1.0656	8.9	22.5	1.0945	12.4
3.1	1.0121	1.7	9.6	1.0384	5.3	16.1	1.0660	8.9	22.6	1.0950	12.5
3.2	1.0125	1.8	9.7	1.0388	5.4	16.2	1.0665	9.0	22.7	1.0954	12.55
3.3	1.0129	1.8	9.8	1.0393	5.4	16.3	1.0669	9.0	22.8	1.0959	12.6
3.4	1.0133	1.9	9.9	1.0397	5.5	16.4	1.0674	9.1	22.9	1.0964	12.7
3.5	1.0137	1.9	10.0	1.0401	5.55	16.5	1.0678	9.1	23.0	1.0968	12.7
3.6	1.0141	2.0	10.1	1.0405	5.6	16.6	1.0682	9.2	23.1	1.0973	12.8
3.7	1.0145	2.0	10.2	1.0409	5.7	16.7	1.0687	9.25	23.2	1.0977	12.8
3.8	1.0149	2.1	10.3	1.0413	5.7	16.8	1.0691	9.3	23.3	1.0982	12.9
3.9	1.0153	2.2	10.4	1.0418	5.8	16.9	1.0695	9.4	23.4	1.0986	12.9
4.0	1.0157	2.2	10.5	1.0422	5.8	17.0	1.0700	9.4	23.5	1.0991	13.0
4.1	1.0161	2.3	10.6	1.0426	5.9	17.1	1.0704	9.5	23.6	1.0996	13.0
4.2	1.0165	2.3	10.7	1.0430	5.9	17.2	1.0709	9.5	23.7	1.1000	13.1
4.3	1.0169	2.4	10.8	1.0434	6.0	17.3	1.0713	9.6	23.8	1.1005	13.15
4.4	1.0173	2.4	10.9	1.0439	6.05	17.4	1.0717	9.6	23.9	1.1009	13.2
4.5	1.0177	2.5	11.0	1.0443	6.1	17.5	1.0722	9.7	24.0	1.1014	13.3
4.6	1.0181	2.6	11.1	1.0447	6.2	17.6	1.0726	9.75	24.1	1.1019	13.3
4.7	1.0185	2.6	11.2	1.0451	6.2	17.7	1.0730	9.8	24.2	1.1023	13.4
4.8	1.0189	2.7	11.3	1.0455	6.3	17.8	1.0735	9.9	24.3	1.1028	13.4
4.9	1.0193	2.7	11.4	1.0459	6.3	17.9	1.0739	9.9	24.4	1.1032	13.5
5.0	1.0197	2.8	11.5	1.0464	6.4	18.0	1.0744	10.0	24.5	1.1037	13.5
5.1	1.0201	2.8	11.6	1.0468	6.4	18.1	1.0748	10.0	24.6	1.1042	13.6
5.2	1.0205	2.9	11.7	1.0472	6.5	18.2	1.0753	10.1	24.7	1.1046	13.6
5.3	1.0209	2.9	11.8	1.0476	6.55	18.3	1.0757	10.1	24.8	1.1051	13.7
5.4	1.0213	3.0	11.9	1.0481	6.6	18.4	1.0761	10.2	24.9	1.1056	13.75
5.5	1.0217	3.0	12.0	1.0485	6.7	18.5	1.0766	10.2	25.0	1.1060	13.8
5.6	1.0221	3.1	12.1	1.0489	6.7	18.6	1.0770	10.3	25.1	1.1065	13.9
5.7	1.0225	3.2	12.2	1.0493	6.8	18.7	1.0775	10.35	25.2	1.1070	13.9
5.8	1.0229	3.2	12.3	1.0497	6.8	18.8	1.0779	10.4	25.3	1.1074	14.0
5.9	1.0233	3.3	12.4	1.0502	6.9	18.9	1.0783	10.5	25.4	1.1079	14.0
6.0	1.0237	3.3	12.5	1.0506	6.9	19.0	1.0788	10.5	25.5	1.1083	14.1
6.1	1.0241	3.4	12.6	1.0510	7.0	19.1	1.0792	10.6	25.6	1.1088	14.1
6.2	1.0245	3.4	12.7	1.0514	7.05	19.2	1.0797	10.6	25.7	1.1093	14.2
6.3	1.0249	3.5	12.8	1.0519	7.1	19.3	1.0801	10.7	25.8	1.1097	14.2
6.4	1.0253	3.6	12.9	1.0523	7.2	19.4	1.0806	10.7	25.9	1.1102	14.3
6.5	1.0257	3.6	13.0	1.0527	7.2	19.5	1.0810	10.8	26.0	1.1107	14.35

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
26.1	1.1111	14.4	32.6	1.1422	17.9	39.1	1.1748	21.4	45.6	1.2088	24.9
26.2	1.1116	14.5	32.7	1.1427	18.0	39.2	1.1753	21.5	45.7	1.2093	24.9
26.3	1.1121	14.5	32.8	1.1432	18.0	39.3	1.1758	21.5	45.8	1.2099	25.0
26.4	1.1125	14.6	32.9	1.1437	18.1	39.4	1.1763	21.6	45.9	1.2104	25.0
26.5	1.1130	14.6	33.0	1.1442	18.15	39.5	1.1768	21.6	46.0	1.2110	25.1
26.6	1.1135	14.7	33.1	1.1447	18.2	39.6	1.1773	21.7	46.1	1.2115	25.1
26.7	1.1140	14.7	33.2	1.1452	18.25	39.7	1.1778	21.7	46.2	1.2120	25.2
26.8	1.1144	14.8	33.3	1.1457	18.3	39.8	1.1784	21.8	46.3	1.2126	25.2
26.9	1.1149	14.8	33.4	1.1462	18.4	39.9	1.1789	21.85	46.4	1.2131	25.3
27.0	1.1154	14.9	33.5	1.1466	18.4	40.0	1.1794	21.9	46.5	1.2136	25.35
27.1	1.1158	14.9	33.6	1.1471	18.5	40.1	1.1799	22.0	46.6	1.2142	25.4
27.2	1.1163	15.0	33.7	1.1476	18.5	40.2	1.1804	22.0	46.7	1.2147	25.45
27.3	1.1168	15.1	33.8	1.1481	18.6	40.3	1.1809	22.1	46.8	1.2153	25.5
27.4	1.1172	15.1	33.9	1.1486	18.6	40.4	1.1815	22.1	46.9	1.2158	25.6
27.5	1.1177	15.2	34.0	1.1491	18.7	40.5	1.1820	22.2	47.0	1.2163	25.6
27.6	1.1182	15.2	34.1	1.1496	18.7	40.6	1.1825	22.2	47.1	1.2169	25.7
27.7	1.1187	15.3	34.2	1.1501	18.8	40.7	1.1830	22.3	47.2	1.2174	25.7
27.8	1.1191	15.3	34.3	1.1506	18.85	40.8	1.1835	22.3	47.3	1.2180	25.8
27.9	1.1196	15.4	34.4	1.1511	18.9	40.9	1.1840	22.4	47.4	1.2185	25.8
28.0	1.1201	15.4	34.5	1.1516	18.95	41.0	1.1846	22.4	47.5	1.2191	25.9
28.1	1.1206	15.5	34.6	1.1521	19.0	41.1	1.1851	22.5	47.6	1.2196	25.9
28.2	1.1210	15.55	34.7	1.1526	19.1	41.2	1.1856	22.5	47.7	1.2201	26.0
28.3	1.1215	15.6	34.8	1.1531	19.1	41.3	1.1861	22.6	47.8	1.2207	26.0
28.4	1.1220	15.7	34.9	1.1536	19.2	41.4	1.1866	22.65	47.9	1.2212	26.1
28.5	1.1225	15.7	35.0	1.1541	19.2	41.5	1.1872	22.7	48.0	1.2218	26.1
28.6	1.1229	15.8	35.1	1.1546	19.3	41.6	1.1877	22.75	48.1	1.2223	26.2
28.7	1.1234	15.8	35.2	1.1551	19.3	41.7	1.1882	22.8	48.2	1.2229	26.2
28.8	1.1239	15.9	35.3	1.1556	19.4	41.8	1.1887	22.9	48.3	1.2234	26.3
28.9	1.1244	15.9	35.4	1.1561	19.4	41.9	1.1892	22.9	48.4	1.2240	26.35
29.0	1.1248	16.0	35.5	1.1566	19.5	42.0	1.1898	23.0	48.5	1.2245	26.4
29.1	1.1253	16.0	35.6	1.1571	19.55	42.1	1.1903	23.0	48.6	1.2250	26.45
29.2	1.1258	16.1	35.7	1.1576	19.6	42.2	1.1908	23.1	48.7	1.2256	26.5
29.3	1.1263	16.1	35.8	1.1581	19.65	42.3	1.1913	23.1	48.8	1.2261	26.6
29.4	1.1267	16.2	35.9	1.1586	19.7	42.4	1.1919	23.2	48.9	1.2267	26.6
29.5	1.1272	16.25	36.0	1.1591	19.8	42.5	1.1924	23.2	49.0	1.2272	26.7
29.6	1.1277	16.3	36.1	1.1596	19.8	42.6	1.1929	23.3	49.1	1.2278	26.7
29.7	1.1282	16.4	36.2	1.1601	19.9	42.7	1.1934	23.3	49.2	1.2283	26.8
29.8	1.1287	16.4	36.3	1.1606	19.9	42.8	1.1940	23.4	49.3	1.2289	26.8
29.9	1.1291	16.5	36.4	1.1611	20.0	42.9	1.1945	23.45	49.4	1.2294	26.9
30.0	1.1296	16.5	36.5	1.1616	20.0	43.0	1.1950	23.5	49.5	1.2300	26.9
30.1	1.1301	16.6	36.6	1.1621	20.1	43.1	1.1955	23.55	49.6	1.2305	27.0
30.2	1.1306	16.6	36.7	1.1626	20.1	43.2	1.1961	23.6	49.7	1.2311	27.0
30.3	1.1311	16.7	36.8	1.1631	20.2	43.3	1.1966	23.7	49.8	1.2316	27.1
30.4	1.1315	16.7	36.9	1.1636	20.2	43.4	1.1971	23.7	49.9	1.2322	27.1
30.5	1.1320	16.8	37.0	1.1641	20.3	43.5	1.1976	23.8	50.0	1.2327	27.2
30.6	1.1325	16.85	37.1	1.1646	20.35	43.6	1.1982	23.8	50.1	1.2333	27.2
30.7	1.1330	16.9	37.2	1.1651	20.4	43.7	1.1987	23.9	50.2	1.2338	27.3
30.8	1.1335	17.0	37.3	1.1656	20.5	43.8	1.1992	23.9	50.3	1.2344	27.3
30.9	1.1340	17.0	37.4	1.1661	20.5	43.9	1.1998	24.0	50.4	1.2349	27.4
31.0	1.1344	17.1	37.5	1.1666	20.6	44.0	1.2003	24.0	50.5	1.2355	27.45
31.1	1.1349	17.1	37.6	1.1671	20.6	44.1	1.2008	24.1	50.6	1.2361	27.5
31.2	1.1354	17.2	37.7	1.1676	20.7	44.2	1.2013	24.1	50.7	1.2366	27.55
31.3	1.1359	17.2	37.8	1.1681	20.7	44.3	1.2019	24.2	50.8	1.2372	27.6
31.4	1.1364	17.3	37.9	1.1686	20.8	44.4	1.2024	24.2	50.9	1.2377	27.7
31.5	1.1369	17.3	38.0	1.1692	20.8	44.5	1.2029	24.3	51.0	1.2383	27.7
31.6	1.1374	17.4	38.1	1.1697	20.9	44.6	1.2035	24.35	51.1	1.2388	27.8
31.7	1.1378	17.4	38.2	1.1702	20.9	44.7	1.2040	24.4	51.2	1.2394	27.8
31.8	1.1383	17.5	38.3	1.1707	21.0	44.8	1.2045	24.45	51.3	1.2399	27.9
31.9	1.1388	17.55	38.4	1.1712	21.05	44.9	1.2051	24.5	51.4	1.2405	27.9
32.0	1.1393	17.6	38.5	1.1717	21.1	45.0	1.2056	24.6	51.5	1.2411	28.0
32.1	1.1398	17.7	38.6	1.1722	21.15	45.1	1.2061	24.6	51.6	1.2416	28.0
32.2	1.1403	17.7	38.7	1.1727	21.2	45.2	1.2067	24.7	51.7	1.2422	28.1
32.3	1.1408	17.8	38.8	1.1732	21.3	45.3	1.2072	24.7	51.8	1.2427	28.1
32.4	1.1412	17.8	38.9	1.1737	21.3	45.4	1.2077	24.8	51.9	1.2433	28.2
32.5	1.1417	17.9	39.0	1.1743	21.4	45.5	1.2083	24.8	52.0	1.2439	28.2

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
52.1	1.2444	28.3	58.6	1.2816	31.6	65.1	1.3205	34.95	71.6	1.3610	38.2
52.2	1.2450	28.3	58.7	1.2822	31.7	65.2	1.3211	35.0	71.7	1.3616	38.2
52.3	1.2455	28.4	58.8	1.2828	31.7	65.3	1.3217	35.05	71.8	1.3623	38.2
52.4	1.2461	28.4	58.9	1.2834	31.8	65.4	1.3223	35.1	71.9	1.3629	38.3
52.5	1.2467	28.5	59.0	1.2840	31.85	65.5	1.3229	35.15	72.0	1.3635	38.3
52.6	1.2472	28.5	59.1	1.2845	31.9	65.6	1.3235	35.2	72.1	1.3642	38.4
52.7	1.2478	28.6	59.2	1.2851	31.95	65.7	1.3241	35.25	72.2	1.3648	38.4
52.8	1.2483	28.65	59.3	1.2857	32.0	65.8	1.3247	35.3	72.3	1.3655	38.5
52.9	1.2489	28.7	59.4	1.2863	32.05	65.9	1.3253	35.35	72.4	1.3661	38.5
53.0	1.2495	28.75	59.5	1.2869	32.1	66.0	1.3260	35.4	72.5	1.3667	38.6
53.1	1.2500	28.8	59.6	1.2875	32.15	66.1	1.3266	35.4	72.6	1.3674	38.6
53.2	1.2506	28.85	59.7	1.2881	32.2	66.2	1.3272	35.5	72.7	1.3680	38.7
53.3	1.2512	28.9	59.8	1.2887	32.3	66.3	1.3278	35.5	72.8	1.3687	38.7
53.4	1.2517	28.9	59.9	1.2893	32.3	66.4	1.3285	35.6	72.9	1.3693	38.8
53.5	1.2523	29.0	60.0	1.2898	32.4	66.5	1.3291	35.6	73.0	1.3699	38.8
53.6	1.2529	29.1	60.1	1.2904	32.4	66.6	1.3297	35.7	73.1	1.3705	38.9
53.7	1.2534	29.1	60.2	1.2910	32.5	66.7	1.3303	35.7	73.2	1.3712	38.9
53.8	1.2540	29.2	60.3	1.2916	32.5	66.8	1.3309	35.8	73.3	1.3719	39.0
53.9	1.2546	29.2	60.4	1.2922	32.6	66.9	1.3315	35.8	73.4	1.3725	39.0
54.0	1.2551	29.3	60.5	1.2928	32.6	67.0	1.3322	35.9	73.5	1.3732	39.1
54.1	1.2557	29.3	60.6	1.2934	32.7	67.1	1.3327	35.9	73.6	1.3738	39.1
54.2	1.2563	29.4	60.7	1.2940	32.7	67.2	1.3334	36.0	73.7	1.3745	39.2
54.3	1.2568	29.4	60.8	1.2946	32.8	67.3	1.3340	36.0	73.8	1.3751	39.2
54.4	1.2574	29.5	60.9	1.2952	32.8	67.4	1.3346	36.1	73.9	1.3757	39.3
54.5	1.2580	29.5	61.0	1.2958	32.9	67.5	1.3352	36.1	74.0	1.3764	39.3
54.6	1.2585	29.6	61.1	1.2964	32.9	67.6	1.3359	36.2	74.1	1.3770	39.4
54.7	1.2591	29.6	61.2	1.2970	33.0	67.7	1.3365	36.2	74.2	1.3777	39.4
54.8	1.2597	29.7	61.3	1.2975	33.0	67.8	1.3371	36.3	74.3	1.3783	39.5
54.9	1.2602	29.7	61.4	1.2981	33.1	67.9	1.3377	36.3	74.4	1.3790	39.5
55.0	1.2608	29.8	61.5	1.2987	33.1	68.0	1.3384	36.4	74.5	1.3796	39.6
55.1	1.2614	29.8	61.6	1.2993	33.2	68.1	1.3390	36.4	74.6	1.3803	39.6
55.2	1.2620	29.9	61.7	1.2999	33.2	68.2	1.3396	36.5	74.7	1.3809	39.7
55.3	1.2625	29.9	61.8	1.3005	33.3	68.3	1.3402	36.5	74.8	1.3816	39.7
55.4	1.2631	30.0	61.9	1.3011	33.3	68.4	1.3408	36.6	74.9	1.3822	39.8
55.5	1.2637	30.05	62.0	1.3017	33.4	68.5	1.3415	36.6	75.0	1.3828	39.8
55.6	1.2642	30.1	62.1	1.3023	33.4	68.6	1.3421	36.7	75.1	1.3835	39.9
55.7	1.2648	30.15	62.2	1.3029	33.5	68.7	1.3427	36.7	75.2	1.3842	39.9
55.8	1.2654	30.2	62.3	1.3035	33.5	68.8	1.3433	36.8	75.3	1.3848	40.0
55.9	1.2660	30.25	62.4	1.3041	33.6	68.9	1.3440	36.8	75.4	1.3855	40.0
56.0	1.2665	30.3	62.5	1.3047	33.6	69.0	1.3446	36.9	75.5	1.3861	40.1
56.1	1.2671	30.4	62.6	1.3053	33.7	69.1	1.3452	36.9	75.6	1.3868	40.1
56.2	1.2677	30.4	62.7	1.3059	33.7	69.2	1.3458	37.0	75.7	1.3874	40.2
56.3	1.2683	30.5	62.8	1.3065	33.8	69.3	1.3465	37.0	75.8	1.3880	40.2
56.4	1.2688	30.5	62.9	1.3071	33.8	69.4	1.3471	37.1	75.9	1.3887	40.3
56.5	1.2694	30.6	63.0	1.3077	33.9	69.5	1.3477	37.1	76.0	1.3894	40.3
56.6	1.2700	30.6	63.1	1.3083	33.9	69.6	1.3484	37.2	76.1	1.3900	40.4
56.7	1.2706	30.7	63.2	1.3089	34.0	69.7	1.3490	37.2	76.2	1.3907	40.4
56.8	1.2712	30.7	63.3	1.3095	34.0	69.8	1.3496	37.3	76.3	1.3913	40.5
56.9	1.2717	30.8	63.4	1.3101	34.1	69.9	1.3502	37.3	76.4	1.3920	40.5
57.0	1.2723	30.8	63.5	1.3107	34.1	70.0	1.3509	37.4	76.5	1.3926	40.6
57.1	1.2729	30.9	63.6	1.3113	34.2	70.1	1.3515	37.4	76.6	1.3933	40.6
57.2	1.2735	30.9	63.7	1.3119	34.2	70.2	1.3521	37.5	76.7	1.3940	40.7
57.3	1.2740	31.0	63.8	1.3126	34.3	70.3	1.3528	37.5	76.8	1.3946	40.7
57.4	1.2746	31.0	63.9	1.3132	34.3	70.4	1.3534	37.6	76.9	1.3953	40.8
57.5	1.2752	31.1	64.0	1.3138	34.4	70.5	1.3540	37.6	77.0	1.3959	40.8
57.6	1.2758	31.1	64.1	1.3144	34.4	70.6	1.3546	37.7	77.1	1.3966	40.8
57.7	1.2764	31.2	64.2	1.3150	34.5	70.7	1.3553	37.7	77.2	1.3972	40.9
57.8	1.2769	31.2	64.3	1.3156	34.5	70.8	1.3559	37.8	77.3	1.3979	41.0
57.9	1.2775	31.3	64.4	1.3162	34.6	70.9	1.3565	37.8	77.4	1.3986	41.0
58.0	1.2781	31.3	64.5	1.3168	34.6	71.0	1.3572	37.9	77.5	1.3992	41.0
58.1	1.2787	31.4	64.6	1.3174	34.7	71.1	1.3578	37.9	77.6	1.3999	41.1
58.2	1.2793	31.4	64.7	1.3180	34.7	71.2	1.3585	38.0	77.7	1.4005	41.1
58.3	1.2799	31.5	64.8	1.3186	34.8	71.3	1.3591	38.0	77.8	1.4012	41.2
58.4	1.2804	31.5	64.9	1.3192	34.8	71.4	1.3597	38.1	77.9	1.4019	41.2
58.5	1.2810	31.6	65.0	1.3198	34.9	71.5	1.3604	38.1	78.0	1.4025	41.3

TABLE VI.—*Relation of brix, specific gravity, and Baumé—Continued.*

Per cent of sugar	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.	Per cent of sugar.	Specific gravity.	Degree Baumé.
78.1	1.4032	41.3	80.1	1.4165	42.3	82.1	1.4300	43.3	84.1	1.4437	44.2
78.2	1.4039	41.4	80.2	1.4172	42.3	82.2	1.4307	43.3	84.2	1.4443	44.3
78.3	1.4045	41.4	80.3	1.4179	42.4	82.3	1.4314	43.4	84.3	1.4450	44.3
78.4	1.4052	41.5	80.4	1.4185	42.4	82.4	1.4320	43.4	84.4	1.4457	44.3
78.5	1.4058	41.5	80.5	1.4192	42.5	82.5	1.4327	43.5	84.5	1.4464	44.4
78.6	1.4065	41.6	80.6	1.4199	42.5	82.6	1.4334	43.5	84.6	1.4471	44.4
78.7	1.4072	41.6	80.7	1.4205	42.6	82.7	1.4341	43.5	84.7	1.4478	44.5
78.8	1.4078	41.7	80.8	1.4212	42.6	82.8	1.4348	43.6	84.8	1.4485	44.5
78.9	1.4085	41.7	80.9	1.4219	42.7	82.9	1.4354	43.6	84.9	1.4492	44.6
79.0	1.4092	41.8	81.0	1.4226	42.7	83.0	1.4361	43.7	85.0	1.4498	44.6
79.1	1.4098	41.8	81.1	1.4232	42.8	83.1	1.4368	43.7	85.1	1.4505	44.7
79.2	1.4105	41.9	81.2	1.4239	42.8	83.2	1.4375	43.8	85.2	1.4512	44.7
79.3	1.4112	41.9	81.3	1.4246	42.9	83.3	1.4382	43.8	85.3	1.4519	44.8
79.4	1.4119	42.0	81.4	1.4253	42.9	83.4	1.4388	43.9	85.4	1.4526	44.8
79.5	1.4125	42.0	81.5	1.4259	43.0	83.5	1.4395	43.9	85.5	1.4533	44.9
79.6	1.4132	42.1	81.6	1.4266	43.0	83.6	1.4402	44.0	85.6	1.4540	44.9
79.7	1.4138	42.1	81.7	1.4273	43.1	83.7	1.4409	44.0	85.7	1.4547	45.0
79.8	1.4145	42.2	81.8	1.4280	43.1	83.8	1.4416	44.1	85.8	1.4554	45.0
79.9	1.4152	42.2	81.9	1.4287	43.2	83.9	1.4423	44.1	85.9	1.4561	45.1
80.0	1.4158	42.2	82.0	1.4293	43.2	84.0	1.4430	44.2	86.0	1.4568	45.1

TABLE VII.—*Correction for the readings of Balling's saccharometer, on account of temperature.*

TO BE SUBTRACTED FROM THE DEGREE READ.

Temp.	Per cent of sugar in solution.												
C.	0	5	10	15	20	25	30	35	40	50	60	70	75
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	.12	.15	.16	.17	.18	.19	.21	.22	.22	.23	.26	.28	.32
15	.09	.11	.12	.14	.14	.15	.16	.17	.16	.17	.19	.21	.25
16	.06	.07	.08	.09	.10	.10	.11	.12	.12	.12	.14	.16	.18
17	.02	.02	.03	.03	.03	.04	.04	.04	.04	.04	.05	.05	.06
TO BE ADDED TO THE DEGREE READ.													
18	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.02
19	.06	.08	.08	.09	.09	.10	.10	.10	.10	.10	.10	.08	.06
20	.11	.14	.15	.17	.17	.18	.18	.18	.19	.19	.18	.15	.11
21	.16	.20	.22	.24	.24	.25	.25	.25	.26	.26	.25	.22	.18
22	.21	.26	.29	.31	.31	.32	.32	.32	.33	.34	.32	.29	.25
23	.27	.32	.35	.37	.38	.39	.39	.39	.40	.42	.39	.36	.33
24	.32	.38	.41	.43	.44	.46	.46	.47	.47	.50	.46	.43	.40
25	.37	.44	.47	.49	.51	.53	.54	.55	.55	.58	.54	.51	.48
26	.43	.50	.54	.56	.58	.60	.61	.62	.62	.66	.62	.58	.55
27	.49	.57	.61	.63	.65	.68	.68	.69	.70	.74	.70	.65	.62
28	.56	.64	.68	.70	.72	.76	.76	.78	.78	.82	.78	.72	.70
29	.63	.71	.75	.78	.79	.84	.84	.86	.86	.90	.86	.80	.78
30	.70	.78	.82	.87	.87	.92	.92	.94	.94	.98	.94	.88	.86

TABLE VIII.—*Allihn's table for the determination of dextrose.*

Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.	Milli-grams of cop-per.	Milli-grams of cu-prous oxid.	Milli-grams of dex-trose.
11	12.4	6.6	76	85.6	38.8	141	158.7	71.8	206	231.9	105.8
12	13.5	7.1	77	86.7	39.3	142	159.9	72.3	207	233.0	106.3
13	14.6	7.6	78	87.8	39.8	143	161.0	72.9	208	234.2	106.8
14	15.8	8.1	79	88.9	40.3	144	162.1	73.4	209	235.3	107.4
15	16.9	8.6	80	90.1	40.8	145	163.2	73.9	210	236.4	107.9
16	18.0	9.0	81	91.2	41.3	146	164.4	74.4	211	237.6	108.4
17	19.1	9.5	82	92.3	41.8	147	165.5	74.9	212	238.7	109.0
18	20.3	10.0	83	93.4	42.3	148	166.6	75.5	213	239.8	109.5
19	21.4	10.5	84	94.6	42.8	149	167.7	76.0	214	240.9	110.0
20	22.5	11.0	85	95.7	43.4	150	168.9	76.5	215	242.1	110.6
21	23.6	11.5	86	96.8	43.9	151	170.0	77.0	216	243.2	111.1
22	24.8	12.0	87	97.9	44.4	152	171.1	77.5	217	244.3	111.6
23	25.9	12.5	88	99.1	44.9	153	172.3	78.1	218	245.4	112.1
24	27.0	13.0	89	100.2	45.4	154	173.4	78.6	219	246.6	112.7
25	28.1	13.5	90	101.3	45.9	155	174.5	79.1	220	247.7	113.2
26	29.3	14.0	91	102.4	46.4	156	175.6	79.6	221	248.7	113.7
27	30.4	14.5	92	103.6	46.9	157	176.8	80.1	222	249.9	114.3
28	31.5	15.0	93	104.7	47.4	158	177.9	80.7	223	251.0	114.8
29	32.7	15.5	94	105.8	47.9	159	179.0	81.2	224	252.4	115.3
30	33.8	16.0	95	107.0	48.4	160	180.1	81.7	225	253.3	115.9
31	34.9	16.5	96	108.1	48.9	161	181.3	82.2	226	254.4	116.4
32	36.0	17.0	97	109.2	49.4	162	182.4	82.7	227	255.6	116.9
33	37.2	17.5	98	110.3	49.9	163	183.5	83.3	228	256.7	117.4
34	38.3	18.0	99	111.5	50.4	164	184.6	83.8	229	257.8	118.0
35	39.4	18.5	100	112.6	50.9	165	185.8	84.3	230	258.9	118.5
36	40.5	18.9	101	113.7	51.4	166	186.9	84.8	231	260.1	119.0
37	41.7	19.4	102	114.8	51.9	167	188.0	85.3	232	261.2	119.6
38	42.8	19.9	103	116.0	52.4	168	189.1	85.9	233	262.3	120.1
39	43.9	20.4	104	117.1	52.9	169	190.3	86.4	234	263.4	120.7
40	45.0	20.9	105	118.2	53.5	170	191.4	86.9	235	264.6	121.2
41	46.2	21.4	106	119.3	54.0	171	192.5	87.4	236	265.7	121.7
42	47.3	21.9	107	120.5	54.5	172	193.6	87.9	237	266.8	122.3
43	48.4	22.4	108	121.6	55.0	173	194.8	88.5	238	268.0	122.8
44	49.5	22.9	109	122.7	55.5	174	195.9	89.0	239	269.1	123.4
45	50.7	23.4	110	123.8	56.0	175	197.0	89.5	240	270.2	123.9
46	51.8	23.9	111	125.0	56.5	176	198.1	90.0	241	271.3	124.4
47	52.9	24.4	112	126.1	57.0	177	199.3	90.5	242	272.5	125.0
48	54.0	24.9	113	127.2	57.5	178	200.4	91.1	243	273.6	125.5
49	55.2	25.4	114	128.3	58.0	179	201.5	91.6	244	274.7	126.0
50	56.3	25.9	115	129.6	58.6	180	202.6	92.1	245	275.8	126.6
51	57.4	26.4	116	130.6	59.1	181	203.8	92.6	246	277.0	127.1
52	58.5	26.9	117	131.7	59.6	182	204.9	93.1	247	278.1	127.6
53	59.7	27.4	118	132.8	60.1	183	206.0	93.7	248	279.2	128.1
54	60.8	27.9	119	134.0	60.6	184	207.1	94.2	249	280.3	128.7
55	61.9	28.4	120	135.1	61.1	185	208.3	94.7	250	281.5	129.2
56	63.0	28.8	121	136.2	61.6	186	209.4	95.2	251	282.6	129.7
57	64.2	29.3	122	137.4	62.1	187	210.5	95.7	252	283.7	130.3
58	65.3	29.8	123	138.5	62.6	188	211.7	96.3	253	284.8	130.8
59	66.4	30.3	124	139.6	63.1	189	212.8	96.8	254	286.0	131.4
60	67.6	30.8	125	140.7	63.7	190	213.9	97.3	255	287.1	131.9
61	68.7	31.3	126	141.9	64.2	191	215.0	97.8	256	288.2	132.4
62	69.8	31.8	127	143.0	64.7	192	216.2	98.4	257	289.3	133.0
63	70.9	32.3	128	144.1	65.2	193	217.3	98.9	258	290.5	133.5
64	72.1	32.8	129	145.2	65.7	194	218.4	99.4	259	291.6	134.1
65	73.2	33.3	130	146.4	66.2	195	219.5	100.0	260	292.7	134.6
66	74.3	33.8	131	147.5	66.7	196	220.7	100.5	261	293.8	135.1
67	75.4	34.3	132	148.6	67.2	197	221.8	101.0	262	295.0	135.7
68	76.6	34.8	133	149.7	67.7	198	222.9	101.5	263	296.1	136.2
69	77.7	35.3	134	150.9	68.2	199	224.0	102.0	264	297.2	136.8
70	78.8	35.8	135	152.0	68.8	200	225.2	102.6	265	298.3	137.3
71	79.9	36.3	136	153.1	69.3	201	226.3	103.1	266	299.5	137.8
72	81.1	36.8	137	154.2	69.8	202	227.4	103.7	267	300.6	138.4
73	82.2	37.3	138	155.4	70.3	203	228.5	104.2	268	301.7	138.9
74	83.3	37.8	139	156.5	70.8	204	229.7	104.7	269	302.8	139.5
75	84.4	38.30	140	157.6	71.3	205	230.8	105.3	270	304.0	140.0

TABLE VIII.—*Allihn's table for the determination of dextrose*—Continued.

Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of dextrose.
271	305.1	140.6	321	361.4	168.1	371	417.7	196.3	421	474.0	225.1
272	306.2	141.1	322	362.5	168.6	372	418.8	196.8	422	475.6	225.7
273	307.3	141.7	323	363.7	169.2	373	420.0	197.4	423	476.2	226.3
274	308.5	142.2	324	364.8	169.7	374	421.1	198.0	424	477.4	226.9
275	309.6	142.8	325	365.9	170.3	375	422.2	198.6	425	478.5	227.5
276	310.7	143.3	326	367.0	170.9	376	423.3	199.1	426	479.6	228.0
277	311.9	143.9	327	368.2	171.4	377	424.5	199.7	427	480.7	228.6
278	313.0	144.4	328	369.3	172.0	378	425.6	200.3	428	481.9	229.2
279	314.1	145.0	329	370.4	172.5	379	426.7	200.8	429	483.0	229.8
280	315.2	145.5	330	371.5	173.1	380	427.8	201.4	430	484.1	230.4
281	316.4	146.1	331	372.7	173.7	381	429.0	202.0	431	485.3	231.0
282	317.5	146.6	332	373.8	174.2	382	430.1	202.5	432	486.4	231.6
283	318.6	147.2	333	374.9	174.8	383	431.2	203.1	433	487.5	232.2
284	319.7	147.7	334	376.0	175.3	384	432.3	203.7	434	488.6	232.8
285	320.9	148.3	335	377.2	175.9	385	433.5	204.3	435	489.7	233.4
286	322.0	148.8	336	378.3	176.5	386	434.6	204.8	436	490.9	233.9
287	323.1	149.4	337	379.4	177.0	387	435.7	205.4	437	492.0	234.5
288	324.2	149.9	338	380.5	177.6	388	436.8	206.0	438	493.1	235.1
289	325.4	150.5	339	381.7	178.1	389	438.0	206.5	439	494.3	235.7
290	326.5	151.0	340	382.8	178.7	390	439.1	207.1	440	495.4	236.3
291	327.4	151.6	341	383.9	179.3	391	440.2	207.7	441	496.5	236.9
292	328.7	152.1	342	385.0	179.8	392	441.3	208.3	442	497.6	237.5
293	329.9	152.7	343	386.2	180.4	393	442.4	208.8	443	498.8	238.1
294	331.0	153.2	344	387.3	180.9	394	443.6	209.4	444	499.9	238.7
295	332.1	153.8	345	388.4	181.5	395	444.7	210.0	445	501.0	239.3
296	333.3	154.3	346	389.6	182.1	396	445.9	210.6	446	502.1	239.8
297	334.4	154.9	347	390.7	182.6	397	447.0	211.2	447	503.2	240.4
298	335.5	155.4	348	391.8	183.2	398	448.1	211.7	448	504.4	241.0
299	336.6	156.0	349	392.9	183.7	399	449.2	212.3	449	505.5	241.6
300	337.8	156.5	350	394.0	184.3	400	450.3	212.9	450	506.6	242.2
301	338.9	157.1	351	395.2	184.9	401	451.5	213.5	451	507.8	242.8
302	340.0	157.6	352	396.3	185.4	402	452.6	214.1	452	508.9	243.4
303	341.1	158.2	353	397.4	186.0	403	453.7	214.6	453	510.0	244.0
304	342.3	158.7	354	398.6	186.6	404	454.8	215.2	454	511.1	244.6
305	343.4	159.3	355	399.7	187.2	405	456.0	215.8	455	512.3	245.2
306	344.5	159.8	356	400.8	187.7	406	457.1	216.4	456	513.4	245.7
307	345.6	160.4	357	401.9	188.3	407	458.2	217.0	457	514.5	246.3
308	346.8	160.9	358	403.1	188.9	408	459.4	217.5	458	515.6	246.9
309	347.9	161.5	359	404.2	189.4	409	460.5	218.1	459	516.8	247.5
310	349.0	162.0	360	405.3	190.0	410	461.6	218.7	460	517.9	248.1
311	350.1	162.6	361	406.4	190.6	411	462.7	219.3	461	519.0	248.7
312	351.3	163.1	362	407.6	191.1	412	463.8	219.9	462	520.1	249.3
313	352.4	163.7	363	408.7	191.7	413	465.0	220.4	463	521.3	249.9
314	353.5	164.2	364	409.8	192.3	414	466.1	221.0			
315	354.6	164.8	365	410.9	192.9	415	467.2	221.6			
316	355.8	165.3	366	412.1	193.4	416	468.4	222.2			
317	356.9	165.9	367	413.2	194.0	417	469.5	222.8			
318	358.0	166.4	368	414.3	194.6	418	470.6	223.3			
319	359.1	167.0	369	415.4	195.1	419	471.8	223.9			
320	360.3	167.5	370	416.6	195.7	420	472.9	224.5			

TABLE IX.—*Determination of maltose in beer.*

[According to Wein.]

Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of maltose.	Milli-grams of cop-per.	Milli-grams of cuprous oxid.	Milli-grams of maltose.
31	34.9	26.1	36	40.5	30.5	41	46.2	34.8	46	51.8	39.1
32	36.0	27.0	37	41.7	31.3	42	47.3	35.7	47	52.9	40.0
33	37.2	27.9	38	42.8	32.2	43	48.4	36.5	48	54.0	40.9
34	38.3	28.7	39	43.9	33.1	44	49.5	37.4	49	55.2	41.8
35	39.4	29.6	40	45.0	33.9	45	50.7	38.3	50	56.3	42.6

TABLE IX.—*Determination of maltose in beer*—Continued.

Milli-grams of cop-per.	Milli-grams of cup-rous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cup-rous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cup-rous oxid.	Milli-grams of mal-tose.	Milli-grams of cop-per.	Milli-grams of cup-rous oxid.	Milli-grams of mal-tose.
51	57.4	43.5	116	130.6	100.8	181	203.8	159.2	246	277.0	217.2
52	58.5	44.4	117	131.7	101.7	182	204.9	160.1	247	278.1	218.1
53	59.7	45.2	118	132.8	102.6	183	206.0	160.9	248	279.2	219.0
54	60.8	46.1	119	134.0	103.5	184	207.1	161.8	249	280.3	219.9
55	61.9	47.0	120	135.1	104.4	185	208.3	162.7	250	281.5	220.8
56	63.0	47.8	121	136.2	105.3	186	209.4	163.6	251	282.6	221.7
57	64.2	48.7	122	137.4	106.2	187	210.5	164.5	252	283.7	222.6
58	65.3	49.6	123	138.5	107.1	188	211.7	165.4	253	284.8	223.5
59	66.4	50.4	124	139.6	108.0	189	212.8	166.3	254	286.0	224.4
60	67.6	51.3	125	140.7	108.9	190	213.9	167.2	255	287.1	225.3
61	68.7	52.2	126	141.9	109.8	191	215.0	168.1	256	288.2	226.2
62	69.8	53.1	127	143.0	110.7	192	216.2	169.0	257	289.3	227.1
63	70.9	53.9	128	144.1	111.6	193	217.3	169.8	258	290.5	228.0
64	72.1	54.8	129	145.2	112.5	194	218.4	170.7	259	291.6	228.9
65	73.2	55.7	130	146.4	113.4	195	219.5	171.6	260	292.7	229.8
66	74.3	56.6	131	147.5	114.3	196	220.7	172.5	261	293.8	230.7
67	75.4	57.4	132	148.6	115.2	197	221.8	173.4	262	295.0	231.6
68	76.6	58.3	133	149.7	116.1	198	222.9	174.3	263	296.1	232.5
69	77.7	59.2	134	150.9	117.0	199	224.0	175.2	264	297.2	233.4
70	78.8	60.1	135	152.0	117.9	200	225.2	176.1	265	298.3	234.3
71	79.9	61.0	136	153.1	118.8	201	226.3	177.0	266	299.5	235.2
72	81.1	61.8	137	154.2	119.7	202	227.4	177.9	267	300.6	236.1
73	82.2	62.7	138	155.4	120.6	203	228.5	178.7	268	301.7	237.0
74	83.3	63.6	139	156.5	121.5	204	229.7	179.6	269	302.8	237.9
75	84.4	64.5	140	157.6	122.4	205	230.8	180.5	270	304.0	238.8
76	85.6	65.4	141	158.7	123.3	206	231.9	181.4	271	305.1	239.7
77	86.7	66.2	142	159.9	124.2	207	233.0	182.3	272	306.2	240.6
78	87.8	67.1	143	161.0	125.1	208	234.2	183.2	273	307.3	241.5
79	88.9	68.0	144	162.1	126.0	209	235.3	184.1	274	308.5	242.4
80	90.1	68.9	145	163.2	126.9	210	236.4	185.0	275	309.6	243.3
81	91.2	69.7	146	164.4	127.8	211	237.6	185.9	276	310.7	244.2
82	92.3	70.6	147	165.5	128.7	212	238.7	186.8	277	311.9	245.1
83	93.4	71.5	148	166.6	129.6	213	239.8	187.7	278	313.0	246.0
84	94.6	72.4	149	167.7	130.5	214	240.9	188.6	279	314.1	246.9
85	95.7	73.2	150	168.9	131.4	215	242.1	189.5	280	315.2	247.8
86	96.8	74.1	151	170.0	132.3	216	243.2	190.4	281	316.4	248.7
87	97.9	75.0	152	171.1	133.2	217	244.3	191.2	282	317.5	249.6
88	99.1	75.9	153	172.3	134.1	218	245.4	192.1	283	318.6	250.4
89	100.2	76.8	154	173.4	135.0	219	246.6	193.0	284	319.7	251.3
90	101.3	77.7	155	174.5	135.9	220	247.7	193.9	285	320.9	252.2
91	102.4	78.6	156	175.6	136.8	221	248.7	194.8	286	322.0	253.1
92	103.6	79.5	157	176.8	137.7	222	249.9	195.7	287	323.1	254.0
93	104.7	80.3	158	177.9	138.6	223	251.0	196.6	288	324.2	254.9
94	105.8	81.2	159	179.0	139.5	224	252.4	197.5	289	325.4	255.8
95	107.0	82.1	160	180.1	140.4	225	253.3	198.4	290	326.5	256.6
96	108.1	83.0	161	181.3	141.3	226	254.4	199.3	291	327.4	257.5
97	109.2	83.9	162	182.4	142.2	227	255.6	200.2	292	328.7	258.4
98	110.3	84.8	163	183.5	143.1	228	256.7	201.1	293	329.9	259.3
99	111.5	85.7	164	184.6	144.0	229	257.8	202.0	294	331.0	260.2
100	112.6	86.6	165	185.8	144.9	230	258.9	202.9	295	332.1	261.1
101	113.7	87.5	166	186.9	145.8	231	260.1	203.8	296	333.2	262.0
102	114.8	88.4	167	188.0	146.7	232	261.2	204.7	297	334.4	262.8
103	116.0	89.2	168	189.1	147.6	233	262.3	205.6	298	335.5	263.7
104	117.1	90.1	169	190.3	148.5	234	263.4	206.5	299	336.6	264.6
105	118.2	91.0	170	191.4	149.4	235	264.6	207.4	300	337.8	265.5
106	119.3	91.9	171	192.5	150.3	236	265.7	208.3			
107	120.5	92.8	172	193.6	151.2	237	266.8	209.1			
108	121.6	93.7	173	194.8	152.0	238	268.0	210.0			
109	122.7	94.6	174	195.9	152.9	239	269.1	210.9			
110	123.8	95.5	175	197.0	153.8	240	270.2	211.8			
111	125.0	96.4	176	198.1	154.7	241	271.3	212.7			
112	126.1	97.3	177	199.3	155.6	242	272.5	213.6			
113	127.2	98.1	178	200.4	156.5	243	273.6	214.5			
114	128.3	99.0	179	201.5	157.4	244	274.7	215.4			
115	129.6	99.9	180	202.6	158.3	245	275.8	216.3			

TABLE X.—*Per cent of fat and solids not fat in milk.*

[According to Babcock.]

Per cent of fat.	Lactometer readings at 15.6° C.											Per cent of fat.
	26.	27.	28.	29.	30.	31.	32.	33.	34.	35.	36.	
0.0	6.50	6.75	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.75	9.00	0.0
0.1	6.52	6.77	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.77	9.02	0.1
0.2	6.54	6.79	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.79	9.04	0.2
0.3	6.56	6.81	7.06	7.31	7.56	7.81	8.06	8.31	8.56	8.81	9.06	0.3
0.4	6.58	6.83	7.08	7.33	7.58	7.83	8.08	8.33	8.58	8.83	9.08	0.4
0.5	6.60	6.85	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.85	9.10	0.5
0.6	6.62	6.87	7.12	7.37	7.62	7.87	8.12	8.37	8.62	8.87	9.12	0.6
0.7	6.64	6.89	7.14	7.39	7.64	7.89	8.14	8.39	8.64	8.89	9.14	0.7
0.8	6.66	6.91	7.16	7.41	7.66	7.91	8.16	8.41	8.66	8.91	9.16	0.8
0.9	6.68	6.93	7.18	7.43	7.68	7.93	8.18	8.43	8.68	8.93	9.18	0.9
1.0	6.70	6.95	7.20	7.45	7.70	7.95	8.20	8.45	8.70	8.95	9.20	1.0
1.1	6.72	6.97	7.22	7.47	7.72	7.97	8.22	8.47	8.72	8.97	9.22	1.1
1.2	6.74	6.99	7.26	7.49	7.74	7.99	8.24	8.49	8.74	8.99	9.24	1.2
1.3	6.76	7.01	7.24	7.51	7.76	8.01	8.26	8.51	8.76	9.01	9.26	1.3
1.4	6.78	7.03	7.28	7.53	7.78	8.03	8.28	8.53	8.78	9.03	9.28	1.4
1.5	6.80	7.05	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30	1.5
1.6	6.82	7.07	7.32	7.57	7.82	8.07	8.32	8.57	8.82	9.07	9.32	1.6
1.7	6.84	7.09	7.34	7.59	7.84	8.09	8.34	8.59	8.84	9.09	9.34	1.7
1.8	6.86	7.11	7.36	7.61	7.86	8.11	8.36	8.61	8.86	9.11	9.37	1.8
1.9	6.88	7.13	7.38	7.63	7.88	8.13	8.38	8.63	8.88	9.13	9.39	1.9
2.0	6.90	7.15	7.40	7.65	7.90	8.15	8.40	8.66	8.91	9.16	9.41	2.0
2.1	6.92	7.17	7.42	7.67	7.92	8.17	8.42	8.68	8.93	9.18	9.43	2.1
2.2	6.94	7.19	7.44	7.69	7.94	8.19	8.44	8.70	8.95	9.20	9.45	2.2
2.3	6.96	7.21	7.46	7.71	7.96	8.21	8.46	8.72	8.97	9.22	9.47	2.3
2.4	6.98	7.23	7.48	7.73	7.98	8.23	8.48	8.74	8.99	9.24	9.49	2.4
2.5	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.76	9.01	9.26	9.51	2.5
2.6	7.02	7.27	7.52	7.77	8.02	8.27	8.52	8.78	9.03	9.28	9.53	2.6
2.7	7.04	7.29	7.54	7.79	8.04	8.29	8.54	8.80	9.05	9.30	9.55	2.7
2.8	7.06	7.31	7.56	7.81	8.06	8.31	8.57	8.82	9.07	9.32	9.57	2.8
2.9	7.08	7.33	7.58	7.83	8.08	8.33	8.59	8.84	9.09	9.34	9.59	2.9
3.0	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.86	9.11	9.36	9.61	3.0
3.1	7.12	7.37	7.62	7.87	8.12	8.37	8.63	8.88	9.13	9.38	9.64	3.1
3.2	7.14	7.39	7.64	7.89	8.14	8.39	8.65	8.90	9.15	9.41	9.66	3.2
3.3	7.16	7.41	7.66	7.92	8.17	8.42	8.67	8.92	9.18	9.43	9.68	3.3
3.4	7.18	7.43	7.69	7.94	8.19	8.44	8.69	8.94	9.20	9.45	9.70	3.4
3.5	7.20	7.45	7.71	7.96	8.21	8.46	8.71	8.96	9.22	9.47	9.72	3.5
3.6	7.22	7.48	7.73	7.98	8.23	8.48	8.73	8.98	9.24	9.49	9.74	3.6
3.7	7.24	7.50	7.75	8.00	8.25	8.50	8.75	9.00	9.26	9.51	9.76	3.7
3.8	7.26	7.52	7.77	8.02	8.27	8.52	8.77	9.02	9.28	9.53	9.78	3.8
3.9	7.28	7.54	7.79	8.04	8.29	8.54	8.79	9.04	9.30	9.55	9.80	3.9
4.0	7.30	7.56	7.81	8.06	8.31	8.56	8.81	9.06	9.32	9.57	9.83	4.0
4.1	7.32	7.58	7.83	8.08	8.33	8.58	8.83	9.08	9.34	9.59	9.85	4.1
4.2	7.34	7.60	7.85	8.10	8.35	8.60	8.85	9.11	9.36	9.62	9.87	4.2
4.3	7.36	7.62	7.87	8.12	8.37	8.62	8.87	9.13	9.38	9.64	9.89	4.3
4.4	7.38	7.64	7.89	8.14	8.39	8.64	8.89	9.15	9.40	9.66	9.91	4.4
4.5	7.40	7.66	7.91	8.16	8.41	8.66	8.92	9.17	9.42	9.68	9.93	4.5
4.6	7.43	7.68	7.93	8.18	8.43	8.68	8.94	9.19	9.44	9.70	9.95	4.6
4.7	7.45	7.70	7.95	8.20	8.45	8.70	8.96	9.21	9.46	9.72	9.97	4.7
4.8	7.47	7.72	7.97	8.22	8.47	8.72	8.98	9.23	9.48	9.74	9.99	4.8
4.9	7.49	7.74	7.99	8.24	8.49	8.74	9.00	9.25	9.50	9.76	10.01	4.9
5.0	7.51	7.76	8.01	8.26	8.51	8.76	9.02	9.27	9.52	9.78	10.03	5.0
5.1	7.53	7.78	8.03	8.28	8.53	8.79	9.04	9.29	9.54	9.80	10.05	5.1
5.2	7.55	7.80	8.05	8.30	8.55	8.81	9.06	9.31	9.56	9.82	10.07	5.2
5.3	7.57	7.82	8.07	8.32	8.57	8.83	9.08	9.33	9.58	9.84	10.09	5.3
5.4	7.59	7.84	8.09	8.34	8.59	8.85	9.10	9.36	9.61	9.86	10.11	5.4
5.5	7.61	7.86	8.11	8.36	8.61	8.87	9.12	9.38	9.63	9.88	10.13	5.5
5.6	7.63	7.88	8.13	8.39	8.64	8.89	9.15	9.40	9.65	9.90	10.15	5.6
5.7	7.65	7.90	8.15	8.41	8.66	8.91	9.17	9.42	9.67	9.92	10.17	5.7
5.8	7.67	7.92	8.17	8.43	8.68	8.94	9.19	9.44	9.69	9.94	10.19	5.8
5.9	7.69	7.94	8.20	8.45	8.70	8.96	9.21	9.46	9.71	9.96	10.22	5.9
6.0	7.71	7.96	8.22	8.47	8.73	8.98	9.23	9.48	9.73	9.98	10.24	6.0

TABLE XI.—*Atomic weights.*^a

Name.	Sym- bol.	Atomic weight.		Name.	Sym- bol.	Atomic weight.	
		H=1.	O=16.			H=1.	O=16.
Aluminum.....	Al.....	26.9	27.1	Neodymium.....		142.5	143.6
Antimony.....	St.....	119.5	120.4	Nickel.....	Ni.....	58.25	58.70
Arsenic.....	As.....	74.45	75.0	Nitrogen.....	N.....	13.93	14.04
Barium.....	Ba.....	136.4	137.40	Osmium.....	Os.....	189.6	191.0
Bismuth.....	Bi.....	206.5	208.1	Oxygen.....	O.....	15.88	16.000
Boron.....	B.....	10.9	11.0	Palladium.....	Pd.....	106.2	107.0
Bromine.....	Br.....	79.34	79.95	Phosphorus.....	P.....	30.75	31.0
Cadmium.....	Cd.....	111.55	112.4	Platinum.....	Pt.....	193.4	194.9
Cæsium.....	Cs.....	131.9	132.9	Potassium.....	K.....	38.82	39.1
Calcium.....	Ca.....	39.8	40.1	Praseodymium.....		139.4	140.5
Carbon.....	C.....	11.9	12.0	Rhodium.....	Rh.....	102.2	103.0
Cerium.....	Ce.....	138.0	139.0	Rubidium.....	Rb.....	84.75	85.4
Chlorine.....	Cl.....	35.18	35.45	Ruthenium.....	Ru.....	100.9	101.7
Chromium.....	Cr.....	51.7	52.1	Samarium.....	Sm.....	149.2	150.3
Cobalt.....	Co.....	68.55	69.00	Scandium.....	Sc.....	43.8	44.1
Columbium.....	Cb.....	93.0	93.7	Selenium.....	Se.....	78.6	79.2
Copper.....	Cu.....	63.1	63.6	Silicon.....	Si.....	28.2	28.4
Erbium.....	Er.....	164.7	166.0	Silver.....	Ag.....	107.11	107.92
Fluorine.....	F.....	18.9	19.05	Sodium.....	Na.....	22.88	23.05
Gadolinium.....		155.8	157.0	Strontium.....	Sr.....	86.95	87.60
Gallium.....	Ga.....	69.5	70.0	Sulphur.....	S.....	31.83	32.07
Germanium.....	Ge.....	71.9	72.5	Tantalum.....	Ta.....	181.5	182.8
Glucinum.....	Gl.....	9.0	9.1	Tellurium.....	Te.....	126.5	127.52
Gold.....	Au.....	195.7	197.2	Terbium.....	Tb.....	158.8	160.00
Hydrogen.....	H.....	1.000	1.008	Thallium.....	Tl.....	202.61	204.15
Indium.....	In.....	113.1	114.0	Thorium.....	Th.....	230.8	232.6
Iodine.....	I.....	125.89	126.85	Thulium.....	Tu.....	169.4	170.7
Iridium.....	Ir.....	191.7	193.1	Tin.....	Sn.....	118.1	119.0
Iron.....	Fe.....	55.5	55.9	Titanium.....	Ti.....	47.8	48.15
Lanthanum.....	La.....	137.6	138.6	Tungsten.....	W.....	182.5	184.00
Lead.....	Pb.....	205.36	206.92	Uranium.....	U.....	237.8	239.6
Lithium.....	Li.....	6.97	7.03	Vanadium.....	V.....	51.0	51.4
Magnesium.....	Mg.....	24.1	24.3	Ytterbium.....	Yb.....	171.9	173.2
Manganese.....	Mn.....	54.6	55.0	Yttrium.....	Yt.....	88.3	89.0
Mercury.....	Hg.....	198.50	200.0	Zinc.....	Zn.....	64.9	65.4
Molybdenum.....	Mo.....	95.3	96.0	Zirconium.....	Zr.....	89.7	90.4

^aClarke, Jour. Am. Chem. Soc., 1901, 23, 90.

APPENDIX.

As stated in the introduction, the methods given in the body of this report were submitted to about 250 chemists for criticism before they were reported to the association. The replies received were referred to the various authors and later were considered by them jointly at a meeting held on November 13, 14, 1901. All suggestions that those present approved of from their own experience were incorporated in the methods reported to the association and are published in this bulletin.

In this appendix are given extracts from replies containing other suggestions, which, though not adopted, were thought to be valuable and worthy of consideration at the hands of other analysts. At the beginning of each extract are indicated the page and the chapter subdivision of this bulletin containing the matter to which the criticism refers. The comments are by the referee.

MEAT AND MEAT PRODUCTS.

Page 7, 1.—Practical men will sometimes say that this or that beef is cotton-seed-fed or slop-fed, judging simply from the appearance of the fat. No doubt such fats do give different factors which it might be well to take into account.—*B. M. Pilthashy.*

Page 10, 4, 5, and 7 (a).—For the determination of water, ash, fat, and total nitrogen I have dried a weighed amount (50 grams) of finely chopped meat on a tared, flat, porcelain or nickel dish till the weight is approximately constant after it has stood in the atmosphere of the room over night. This is then immediately finely powdered and tightly stoppered, and is used for the above determinations.—*E. E. Smith.*

Page 10, 6.—Two grams of the dried sample seems an unnecessarily large quantity. I have used 0.5 to 1.0 gram. I think it would be well to specify "passed through a 100 (or 80) mesh sieve."—*E. E. Smith.*

Page 12, 7, (f).—It is quite common to determine meat bases directly by the bromin method without previous determinations of proteoses, peptones, and gelatin. How would the results thus obtained compare with those obtained according to (f)? Is the direct estimation of meat bases from the amount of nitrogenous matter not precipitated by bromin incorrect?—*A. P. Bryant.*

Comment by Mr. Bigelow.—Results by Mr. Trescot in this laboratory show a material error in the method suggested by Mr. Bryant, owing to the decomposition of meat bases with the evolution of nitrogen.

Page 16, 13.—I have succeeded best in extracting coloring matter from sausages by maceration with acidulated alcohol, using hydrochloric acid.—*A. S. Mitchell.*

EDIBLE OILS AND FATS.

Page 20, 2.—In preference to the three methods given, I consider the Sprengel-tube method by far the most accurate, as well as the most rapid, process for determining the specific gravity at the boiling point. This is particularly the case when a large number of samples have to be examined together.

I may add that I consider accurate determinations of specific gravity especially

valuable when taken in conjunction with some other measurement (volatile acid, iodine, absorption, etc.). A fairly constant ratio may often be observed between the specific gravity and other measurements for a particular oil. Addition of an adulterant may upset this relationship without, however, bringing the specific gravity (or other measurement) outside the limits for the pure oil.—*Edgar B. Kenrick.*

Page 22, 2, (b), (2).—Why not use Westphal balance to determine specific gravity at temperature of boiling water?—*A. G. Woodman.*

Page 22, 3.—I think it is time that a vigorous protest be made against the practice, now becoming quite common, of reporting refractive indices by purely arbitrary numbers. One would think that the confusion resulting from the use of Twaddell, Baumé, etc., degrees of specific gravity should be sufficient warning.

The omission of any statement as to the optical relation between the readings of the Zeiss instrument and the refractive indices leaves the reader in doubt as to whether the table given is of any use beyond the individual instrument for which it was constructed. The writer of a recent text-book on physical measurements gives at the end of the book a table for the conversion of the readings of the Pulfrich refractometer into refractive indices. No warning is given to the reader that the table will not apply to any Pulfrich refractometer. The table, in fact, is quite useless for the instrument now in use in my laboratory. The index of refraction in the Pulfrich instrument is the square root of the difference between the square of the refractive index of the glass prism and the square of the sine of the angle measured. Since the refractive index of the prism in any particular instrument is not the same as that of the one for which the table in the book is made, it is obvious that the said table can not be used with my instrument.

After an experience of twelve years with two forms of the Pulfrich refractometer, I am able to say that this instrument leaves nothing to be desired in point of accuracy and ease of operation.—*Edgar B. Kenrick.*

Page 25, 4.—In weighing the fat for determination of iodine absorption I use Weston's small, flat-bottomed glass cylinders and a narrow-mouthed bottle. (See our report for 1896, p. 23.) Would it not be well to make the instruction a little more elastic, so as to cover any form of glassware found efficient?—*A. L. Winton.*

Referring to the "wide-mouth bottle," I should prefer small glass stopper on account of greater danger of loss of iodine from large one.—*A. G. Woodman.*

Page 26, 5, (a).—It has always been customary in the laboratory at Munich, Bavaria, under Professor Hilger, to keep the solutions of iodine and mercuric chloride separate. They were mixed in equal proportions forty-eight hours before use.—*Emil Schlichting.*

Page 26, 5, (b) and (c).—Instead of about 5 grams of the melted fat, I think it better to use from 1 to 2 grams. Instead of a reflux condenser, I prefer a small funnel placed in mouth of flask. This is perfectly satisfactory and more convenient.—*A. G. Woodman.*

Page 29, 11.—The method of using 5 grams of fat is satisfactory for most analytical work, but that I may be able to present a sample of the adulterant found as evidence in court, in the case of adulterated linseed oils, it has been my practice to use 10 grams, following the method of Morawski and Demski, page 172 of Benedikt and Lewkowitch (*Oils, Fats, and Waxes*). By this method the removal of the alcohol used in saponification is avoided, as is also to a great extent the solubility of the alkaline soap solution in ether.

Ten grams of fat are saponified in a flask with 5 grams of caustic potash dissolved

in the least amount of water and 50 cc of stronger alcohol. The mixture is boiled for half an hour with inverted condenser. (I have found saponification by this method insufficient in the case of waxes, notably beeswax.) After saponification 50 cc of hot water are added, the mixture is cooled, and shaken out with petroleum ether.

Page 30, 12.—Commenting upon this method, I will state that while Wiley's method is undoubtedly the most accurate for determination of the melting point, the capillary-tube method is so much more convenient that most of the chemists in this section use it in their commercial work. I would suggest that that method be reinstated as an alternate method.—*A. S. Mitchell.*

Page 32, 14.—The directions for the Maumené figure are doubtless designed especially for olive oil; but inasmuch as cotton-seed, sesame, or poppy seed and the like might be included among edible oils, would it not be well to consider whether "strongest" sulphuric acid can be added to the latter oils without such frothing and evolution of SO_2 as would vitiate the results? Some experiments on the Maumené test have recently been made in this laboratory^a. The details are now being prepared for publication.—*H. C. Sherman.*

Comment by Mr. Tolman.—This suggestion regarding the method for determining the Maumené figure is very appropriate and it will be necessary to modify the method when working with such oils as are mentioned above. Sherman^a uses an acid which will give a rise of temperature with water of 33°–34° C., and calculates the "specific temperature reaction" as given in the methods for oil analysis. But he finds that slightly lower results are obtained by this method. Each analyst should determine the constants for the conditions under which he is working.

Page 33, 17.—I should prefer to use the fatty acids for the Bechi test rather than the oil or fat itself.—*A. G. Woodman.*

DAIRY PRODUCTS.

Page 35, 2.—The dish used should be platinum or light porcelain. I should bar the use of either aluminum or tin. They are too easily subject to corrosion and change of weight. A small rod, about 1 inch by 3 or 4 millimeters, should be weighed with the dish, and the milk and sand should be stirred up when milk is added. This will greatly facilitate drying and no crust will form on top of the sand. In this manner more milk can be advantageously used, and I should advise increasing the amount to an optional 5 or even 10 cc. Anyone using the rod once will never after do without it.—*Charles L. Parsons.*

Page 36, 6.—From the expression, "if the milk contains one or more parts per 10,000 of formaldehyde," one would gather that the test will detect any amount of formaldehyde greater than one part per 10,000, whereas I believe that if the amount of formaldehyde be increased above a certain point the test will no longer show it.—*A. G. Woodman.*

Page 36, 6.—I think the test with phloroglucinol by far the easiest of use accompanied by certain identification, and should be given direct.—*Charles L. Parsons.*

Page 36, 6.—Under the detection of preservatives, I do not think it well to give the sulphuric acid test because there is such a variation between different chemists as to the reliability of this method and the manner of operating it. I would suggest the hydrochloric acid with ferric chlorid as more satisfactory and reliable, and its use is confined to the detection of formaldehyde in milk.

^a "Science," 1901, p. 30.

Page 38, 7.—Under special tests for process butter I believe the appearance and qualitative tests of the curd are very valuable and should be given. *—R. E. Doolittle.*

Page 38, 7.—In the examination of renovated butter by the Reichert process it has been my experience that where Leffman's glycerol method is used the results are uniformly lower than where saponification is effected with alcohol under pressure. It is also my experience that renovated butter when saponified, as is my habit, with alcohol, gives Reichert figures upon 5 grams of fat of from 26 to 28.5. Creamery butter, under these circumstances, gives slightly higher figures, ranging from 28 to 32 and very rarely higher. In other words, I find renovated butter to run upon an average two points lower than creamery butter.

Page 39, 9.—I am of the opinion that complete methods should be given for the detection of coloring matters in butter and its substitutes. This is one of the most important subjects that is engaging the attention of food chemists at the present time, because of the anticolor oleomargarine laws which have been enacted in the several States. I see the referee on Coloring Matter does not take up the subject of colors. In my work I make a preliminary test as follows: Take two portions of about 2 grams or more (according to apparent depth of color) of the filtered fat and dissolve each in a separate tube with ordinary ether. To one test tube add 1 or 2 cc. of dilute hydrochloric acid and to the other about the same quantity of dilute potassium hydroxid solution; shake both well and let stand. If the commonly used azo-dye be present, the first test tube will give a bright pink to deep wine-red color, according to amount of coloring matter present to the acid solution, while the potash solution of the second tube will be uncolored. On the other hand, if annatto be present the potash solution will be colored yellow, varying in depth with amount present, while the acid solution of the first tube will be uncolored. Having thus found the class the color probably belongs to, I can supply the different tests for that particular class.

For the azo color used in butter I would call attention to method of J. F. Geisler.^b This is a very satisfactory and reliable method. For the vegetable colors, such as annatto, I think the methods of Martin and Cornwall should both be given complete.

Page 40, 3.—I would suggest that in my experience it has generally been sufficient to grind the cheese and place it in a muslin strainer upon the water bath for a short time, whereupon sufficient fat will be run out and may be filtered and dried. The addition of chemicals is thus wholly avoided.—*A. S. Mitchell.*

SPICES.

Page 55, 1.—To detect stems in ground cloves, shake up a little of the material in a test tube with alcohol (or other convenient solvent), allow the sediment to settle, and examine the absorption spectrum of the tincture for the characteristic bands of chlorophyll. It would appear that the green coloring matter is absent from the clove buds, while the stems contain it in considerable quantity.—*Edgar B. Kenrick.*

Page 55, 3.—I would recommend the determination of moisture (adventitious), as distinguished from volatile oil, by exposure in vacuo over colorless sulphuric acid. The moisture escapes before the volatile oil is appreciably volatilized. The marked discoloration of the acid may be taken as indicating the point at which notable quantities of volatile oil begin to come off. (See my work on cloves in Bul. 73.)—*A. McGill.*

Comment by Mr. Winton.—Certainly a promising method with obvious advantages, which should be studied by the association. Our standards are based, however, on analyses made by another method.

^aJour. Am. Chem. Soc., 1902, 22, 150.

^bJour. Am. Chem. Soc., 1898, 20, 110.

Page 55, 3.—I would suggest temperature of water bath, say about 100° instead of 110° C. A great many laboratories have only water baths, and the temperature obtainable is only 100° C.—*J. A. Le Clerc.*

Comment by Mr. Winton.—Temperature of 110° , although in some ways not so convenient as 100° , facilitates the removal of the volatile oils.

Page 56, 10.—Most published methods involve the assumption that oil of cloves exerts no vapor pressure at ordinary temperatures. This is very far from being the case. In the process described on page 2, a mixture of ether and oil of cloves will lose both ether and oil of cloves when allowed to "evaporate at room temperature." A second loss will take place in standing eighteen hours over sulphuric acid, the sulphuric acid continually absorbing the vapor of the oil as it is given off.

If cloves are finely powdered and exposed to the air in a thin layer, it will be found that after the lapse of a week or two the loss in weight which has taken place will be approximately equal to the amount of volatile oil originally present. The final weight, or rather the weight after the oil has evaporated, will vary slightly with the pressure of water vapor in the atmosphere. The fact that commercial ground cloves often contain very little volatile oil may sometimes be due to the fact that the cloves have been long ground, the oil having escaped by evaporation.

The correct method indicated would seem to be:

(1) Allow one of the volatile constituents to become in equilibrium with a limited atmosphere kept continually saturated with this constituent (e. g., water) at a given temperature.

(2) Allow the other volatile constituent (oil) to evaporate completely into an unlimited atmosphere free from this constituent.

(3) Restore the original conditions in (1).

The difference in weight between (1) and (3) will give the weight of the second constituent.

(All the oil and water may be driven off by a few hours' heating in a water oven.)

I have used various modifications of the above principle, but have so far had no opportunity of checking their absolute accuracy, as all published methods seem to me wrong in principle for the reason already given.—*E. B. Kenrick.*

Page 56, 10.—A student in this laboratory, Mr. L. L. Watters, last year made some experiments in regard to the determination of oil of sage by a method practically the same as this, except that very light petroleum ether was used for extraction. He found that it was difficult to drive off all of the ether without some of the oil, or to tell when all the solvent was driven off. Also sage oil left a residue, small, but rather variable, when evaporated, and brought to constant weight at 100° C. He thought the amount of residue thus left was influenced by the other constituents of the ether extract. A partial correction was obtained by adding to an aliquot part of the ether solution, before evaporation, a known weight of sage oil and carrying this through the same process as a blank. Possibly it might be worth while to try something of this sort with other volatile oils.—*H. C. Sherman.*

Comment by Mr. Winton.—Our standards are based on ether extraction. The method, we know, is not perfect, but we should go slow in making changes. The points named are worthy of further study.

Page 56, 10.—Could not petroleum ether or gasoline be used alternatively here or wherever extraction with absolute ether is recommended? The latter is better on account of the difficulty of keeping the ether perfectly anhydrous during the extraction.—*A. G. Woodman.*

Comment by Mr. Winton.—Petroleum ether has certain advantages over ether, but among the disadvantages in this instance is the fact that our standards of composition are based on the other method.

Page 56, 10.—Under determination of volatile and nonvolatile ether extracts I think he consumes too much time. He extracts with a continuous apparatus for twenty hours.—*J. A. Wesener.*

Comment by Mr. Winton.—The method for volatile and nonvolatile ether extract takes time, but I have found no satisfactory short cuts. Furthermore, our proposed standards are based on this method.

Page 56, 11.—Alcohol extracts from pepper a large amount (10 to 12 per cent) of matter which is easily volatile from its alcoholic solution, but is not volatile at the same temperature by dry heating. (See Bul. 10, Inland Revenue Laboratory, p. 18.)—*A. McGill.*

Comment by Mr. Winton.—A matter worthy of future study.

Page 57, 12.—The method we are using and one we much prefer to the copper-reducing method for sugars is to conduct the determination similarly to the copper-plating method until the copper suboxid has been dissolved in nitric acid. Then proceed as follows: Add sulphuric acid to displace the excess of nitric acid and boil until all fumes of nitric dioxid have disappeared. Then neutralize with sodium carbonate. Redissolve the precipitated copper in acetic acid, boil to expel excess of that acid, cool, and add enough potassium iodid to change all of the copper to cuprous iodid, with the liberation of free iodine. The free iodine thus liberated is then titrated with deci-normal ammonium thiosulphate, using starch as indicator. This method in our laboratory is both rapid and accurate.—*J. A. Wesener.*

Comment by Mr. Winton.—The association gives choice of several methods for determining of reduced copper, any of which may be employed. Personally, I prefer weighing either the CuO or Cu_2O in a Gooch crucible.

Page 57, 12.—In following Allihn's method, you direct that the solution, after addition of reducing solution, be heated merely to boiling, whereas in the A. O. A. C. version the solution is boiled two minutes.—*W. D. Bigelow.*

Comment by Mr. Winton.—The table used in connection with the Allihn method is based on Allihn's experiments with different quantities of pure dextrose, heating in each case until boiling begins again. So long as we use Allihn's table, we should follow his instructions. The writer, in a long series of experiments^a with both pure starch and dextrose, found that the original Allihn method gave accurate results, whereas longer boiling brought the results too high. The German food analysts use the original Allihn method.

Page 57, 12.—We have found it better to make the copper solution very slightly acid. We include 1 cc of strong sulphuric acid in the 500 cc of copper sulphate solution.—*A. G. Woodman.*

Comment by Mr. Winton.—A subject for study by the association.

Page 58, 14.—Several inquiries have been received regarding the details of the method prescribed for the determination of crude fiber. Paper always gives a clear filtrate and nearly filters rapidly, whereas linen does not always retain the fine material and Gooch crucibles sometimes clog, rendering filtration impossible. A possible disadvantage of a weighed paper is that it may lose weight on treatment with soda, but this loss can be determined by blank experiment and a correction introduced. Of course a Gooch crucible is more convenient, provided it does not clog, but to make its use obligatory renders the method impossible for many samples.—*A. L. Winton.*

Page 58, 14.—In the determination of crude fiber, the centrifuge will be found a

^a Conn. Expt. Sta. Rept., 1897, p. 128; Jour. Anal. Chem., 1888, p. 129.

great help; since filtration after use of alkali is almost impossible, and always most tedious.—*A. McGill.*

Comment by Mr. Winton.—Use of paper obviates the slow filtration.

Page 58, 14.—The only exception I take to the method of analysis as outlined is the method of estimating crude fiber. The official method is always tedious, and usually impossible with spices, owing to clogging of Gooch filter. The method as given is liable to be inaccurate, owing to difficulty of transferring fiber from paper, possibility of removing paper fiber, and the assumption that the weight of filter paper after washing with water, alcohol, and ether, and drying at 100 C.° is the same as before treatment. A method I have used with satisfaction consists in filtering through linen after acid, and again after alkali treatment, then transferring to Gooch crucible and proceeding as in official method.—*E. N. Eaton.*

Comment.—The method I recommend is that of the A. O. A. C., except that the fiber is weighed on a paper filter instead of the Gooch crucible. I can fully agree with Eaton that the "official method is always tedious and usually impossible with spices, owing to the clogging of Gooch filter," but I prefer a paper, both for the acid and alkali filtration. There is little danger of fiber being detached from the paper after the acid filtration.

Page 59, 16.—I think the Jena flasks used in determining nitrogen of nonvolatile ether extract should be described more exactly, whether round or flat-bottomed, length of neck, etc. Would it not be satisfactory to make extraction in the ordinary flask and transfer with water or ether? The flask you prescribe could not be used with the Knorr extraction apparatus.—*W. D. Bigelow.*

Comment by Mr. Winton.—I prefer a flat-bottomed Jena flask, 15 centimeters high, but other shapes and sizes may be used without affecting the result. The extract, after drying, is not easily removed with ether; hence it is strongly advised to digest for nitrogen in the extraction flask, transferring to a larger flask for distillation. The kind of apparatus used is not important, provided the extraction and digestion are complete, and mechanical loss is avoided.

FLAVORING EXTRACTS.

Page 70, 4.—I would suggest the following method for the estimation of vanillin:^a Two cubic centimeters of the vanilla extract is measured into a test tube and sufficient freshly precipitated lead hydroxid added to completely decolorize. The mixture is washed onto a filter and the filtrate and washings collected in a Nessler tube. Bromin water is then added, after which enough of a freshly prepared 10 per cent solution of ferrous sulphate is added to get the maximum bluish-green color that will be produced if vanillin is present.

A standard solution is prepared by dissolving, say 50 mm of pure vanillin in 100 cc of water. A series of standards is then made, taking for instance $\frac{1}{2}$, 1, 2, $2\frac{1}{2}$, 3, etc., cc of the vanillin solution in Nessler tubes, each being treated with 2 or 3 drops of Bromin water, and with ferrous sulphate solution, and made up to the 50-cc mark.

The lead hydroxid is prepared by dissolving 200 grams of lead acetate in 850 cc of water. The solution is filtered and a strong solution of potassium hydroxid is added in excess, and the precipitate is washed thoroughly several times by decantation.^b—*A. E. Leach.*

FERMENTED AND DISTILLED LIQUORS.

Page 83, 6.—When a liquor is titrated and the indicator does not give the color reaction distinctly on account of the color of the liquor after that point where the red goes to violet, the end reaction is gotten by dipping a capillary tube into the

^a Ztschr. anal. Chem., 1894, 33, 242.

^b Ztschr. anal. Chem., 1894, 33, 242; Mass. Board of Health Rept. for 1899, 629.

liquid after every other cubic centimeter and putting the point of it upon a piece of litmus paper. After the absorption of the liquor from the tube the center of the blot will show the alkaline blue on red litmus paper before a drop will. This method was brought before the local section of the American Chemical Society some years ago by Dr. S. Waldbott.—*B. M. Pilhashy.*

Page 84, 10.—Allow me to call attention to a point that is often overlooked in the examination of fermented and distilled liquors. The determination of glucose in wines, etc., is often based on the examination of the sample for dextrine. This in itself is all right except that very little commercial glucose is used in any fermented liquor except beer. The product that is used is grape sugar, which has been so highly converted that it will not give more than a qualitative test for dextrine. The only way to determine the presence of grape sugar is by double polarization before and after inversion. Nor is this positive proof of adulteration since, in some cases, invert sugar is used besides grape sugar, and the amounts are so well balanced that the polarization due to these two products is 0. In cases of this kind the addition of the foreign bodies can only be determined indirectly. The cane sugar is determined by reduction before and after inversion, the difference between the two results being calculated to cane sugar. The direct reduction is then due to the mixture of the grape and invert sugars. A mixture of about one part of grape sugar to to $2\frac{1}{2}$ parts of invert sugar will give a direct polarization, and a polarization after inversion of 0. In some cases I have found that direct polarization was due to the cane sugar added. This is only the case where the fermentation was complete and all the sugar of the grape turned into alcohol.

The point that I want to bring out is that a certain relation exists between the amounts of sugars found by polarization before and after conversion, and the amounts of reducing sugars found by the reduction method before and after conversion, and that the absence of this relation is always proof of adulteration due to the addition of grape or invert sugar or both.—*Edward Gudeman.*

BAKING POWDER AND BAKING-POWDER CHEMICALS.

Page 98, 1.—Regarding the determination of carbon dioxid, I will say that I have used for factory control work a method which requires for its execution a slight modification of the Kjeldahl nitrogen apparatus, receiving the distillate in a solution of sodium hydroxid whose titre of free alkali is known; afterwards titrating with standard acid, using phenacetolin as the indicator. I am satisfied that with the cooperation of the association this method could be made as good for carbon dioxid as the Kjeldahl is for nitrogen.

Figure 7 illustrates the apparatus used by myself for the estimation of carbon dioxid in low-grade phosphate rock, such as is used for fillers in the manufacture of commercial fertilizers.

The flask C is an ordinary globe chemical flask having a capacity of 1,000 cc, fitted with a funnel tube having a stopcock at B. The flask E is an absorption flask having two bulbs, as shown. It should have a capacity of about 200 cc. The condenser D is a Liebig condenser of brass, with inner tube of block tin, as used in the Kjeldahl nitrogen process. This tin tube is connected with the bulb tube of flask C by a bit of rubber tubing and terminates in a glass tube which is fitted by a rubber stopper to flask E.

The determination is made as follows: Place 500 cc of freshly boiled distilled water in flask C; allow it to cool, and, if convenient, reduce its temperature to 20° or lower; then introduce into flask C about 1 gram of the substance to be estimated. Place in funnel tube a quantity of normal sulphuric acid considerably in excess of that required to decompose the substance. Place in the flask E 50 cc of normal or double normal sodium or potassium hydroxid solution. The exact content of hydroxid in this solution must be determined by titrating with decinormal sulphuric acid and phenacetolin previous to using it—in a separate portion, of course.

The apparatus is then connected, as shown in the illustration, with the glass tube dipping in the liquid in flask E. The stopcock B is then partly opened, and the acid is allowed to flow in at such a rate that the bubbles pass through E about one per second. Slowly increase the heat to boiling and distill about 50 cc.

After the distillation, titrate the liquid in E with decinormal sulphuric acid and phenacetolin, or, better, make it up to a definite quantity and take an aliquot part for titration, stopping at the first appearance of the pink. The difference between the amount of hydroxid shown by this last titration and that known to have been contained in it previous to receiving the distillate is the amount of hydroxid that has been converted into carbonate, and from this the quantity of carbon dioxide may readily be calculated. A check on the work may be made by titrating the yellow color, and the difference between the appearance of the pink and the appearance of the yellow is the amount of hydroxid in combination with carbon dioxide.

This method, for use in the determination of available carbon dioxide in baking powder, could be modified by placing the powder in C without water and introducing water gradually through the funnel tube and afterwards heating it as described. —*J. C. Mims.*

Page 100, 1, (a), (3).—It is convenient to introduce sample wrapped in a cartridge in tissue paper into the dry flask. The tissue paper may be colored with litmus or strips of litmus paper may be introduced at the same time. When the baking powder is badly made and contains excess of the acid ingredient this is indicated by the litmus remaining red at end of operation. Usually the sodium carbonate is in excess, and the paper remains blue.

Page 101, 1, (b), (3).—I find that 4 grams of a baking powder may safely and conveniently be employed in determining carbon dioxide. This reduces the factor needed to convert to percentage and conduces to accuracy. With a maximum value for baking powder this amount yields from 0.5 to 0.6 gram of carbon dioxide.

Page 104, 2.—I find it convenient to determine the excess of sodium carbonate occurring in most baking powders by replacing the tubes after determination of the available carbon dioxide, adding acid to the decomposing flask, and continuing the operation without recharging. The carbon dioxide now obtained is that due to excess of sodium bicarbonate. —*A. McGill.*

Page 104, 7.—We desire to make the following preliminary announcement of a method for the application of the polariscope to the estimation of tartaric acid in commercial products:

The commercial products containing tartaric acid fall into three classes, corresponding to the three methods of analysis described below:

Class I.—Tartaric acid and mixtures containing alkaline tartrates and calcium tartrate but no other optically active substances or materials capable of modifying the rotation of tartaric acid in ammoniacal solution (e. g., alum, iron). To this class belong Rochelle salt, potassium tartrate, cream of tartar, calcium tartrate, and many effervescing preparations of the Pharmacopœia.

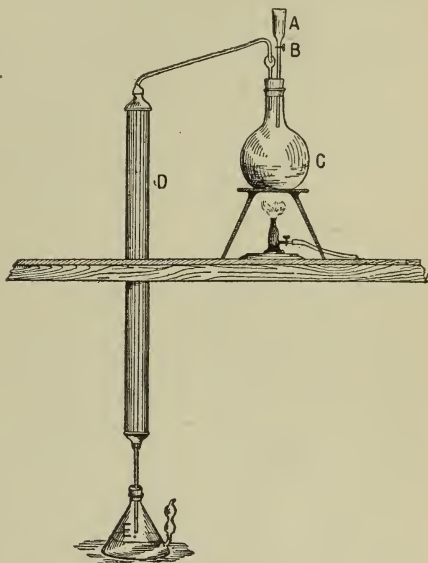


FIG. 7.—Mims' apparatus for the determination of carbon dioxide.

Class II.—Mixtures of the above (I) with sugar. To this class belong many of the effervescing compounds of the Pharmacopœia and most of the similar proprietary preparations.

Class III.—Mixtures of members of Class I with modifying agents and traces of optically active substances. To this class belong mixtures containing alum, those containing traces of iron and aluminum, and those of which starch is a constituent, the latter containing almost invariably traces of active substances soluble in cold water. Consequently all baking powders and mixtures of cream of tartar with cream of tartar substitutes fall into this division.

METHODS OF ANALYSIS.

Class I.—The method employed in the analysis of materials of this group is based on the fact that in the presence of excess of ammonia the rotation of the solution is proportional to the concentration of the tartaric acid, and is independent of the other bases and acids present.

(a) *The substance is completely soluble in dilute ammonia.*—A weighed quantity of the material containing not more than 1 gram tartaric acid is placed in a 25 cc measuring flask, moistened with 3 or 4 cc of water, and conc. ammonia (sp. gr. 0.880) added in quantity sufficient to neutralize all acids that may be present and leave about 1 cc in excess. The actual amount of the excess is not of importance, but a greater quantity than 1 cc of free ammonia should be avoided. The solution is then made up to 25 cc with water, filtered, if necessary, through a dry filter, and measured in a 20 cm tube in the polarimeter.

The amount of tartaric acid ($C_4H_6O_6$) in grams (y) in the material taken is given by the formula:

$$y = 0.00519x$$

where x is the rotation in minutes.

(b) *The substance is not completely soluble in dilute ammonia.*—In this case calcium tartrate is probably present and may be determined as follows: Treat 1 gram of the substance (or an amount containing not more than 1 gram tartaric acid) in a small beaker with 15 cc of water and 10 drops of conc. hydrochloric acid. Heat gently till both the potassium and calcium tartrates have passed into solution, and then, while still hot, add 2 cc of conc. ammonia (or enough to produce an ammoniacal smelling liquid) and about 0.1 gram of sodium phosphate dissolved in a little water. Transfer to a 25 cc measuring flask, cool, make up to the mark with water, filter through a dry filter, and polarize the filtrate in a 20 cm tube. The tartaric acid is calculated from the formula given under (a).

The precipitation of the calcium by means of sodium phosphate is not absolutely necessary, but when this is not done, in cases where the proportion of calcium in the sample is high, there is a great tendency for the calcium tartrate to crystallize out from the ammoniacal solution before the reading is made.

The tartaric acid present as bitartrate of potash may be determined by proceeding as in (a), the calcium tartrate being practically insoluble in cold ammonia solution.

The tartaric acid present as calcium tartrate is given, with sufficient accuracy for most purposes, by the difference between the results of (a) and (b). If more accurate results are required, the residue insoluble in ammonia in (a) may be dissolved in a little hydrochloric acid and treated as above with sodium phosphate and ammonia.

It may be noted that the method given below, under Class III, is applicable to this class also, but in most cases the above procedure will be found simpler.

Class II.—The determination of tartaric acid in substances of this class is an extension of the method given under I. In ammoniacal mixtures containing both tartaric acid and sugar the rotation of each is unaffected by the presence of the other substance, and consequently the rotation of the tartaric acid may be obtained by subtracting from the total rotation that part due to the sugar. The cane sugar may

be conveniently determined by Clerget's method, both readings (before and after inversion), however, being made after addition of ammonia. Should the sugar in these materials have become partly inverted, the reducing sugar must be determined by Fehling's process, and due allowance made for it.

Class III.—Direct readings of rotation in ammoniacal solution are inadmissible in analyses of the substances of this class on account of the influence of iron and aluminum on the rotation of tartaric acid, and on account of the small but unknown rotation of the trace of inverted starch.

Accurate determinations, however, may be made in the presence of excess of ammonium molybdate in neutral solution. The latter substance has the property of greatly increasing the rotation of tartaric acid, so that by its use the small rotation of the inverted starch is made insignificant. It is to be noted, however, that this increased rotation is very sensitive to the presence of alkali and acid, and is, moreover, modified by phosphates. It is therefore necessary, in the first place, to remove the phosphoric acid, and, secondly, to bring the solution to a definite state of neutrality. Both these results are attained by the following procedure, the details of which must be carefully adhered to:

Solutions required.—The following solutions must be prepared, but need not be made up very accurately:

Molybdate solution: 44 grams ammonium heptamolybdate in 250 cc.

Citric acid solution: 50 grams citric acid in 500 cc.

Magnesium sulphate solution: 60 grams $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in 500 cc.

Ammonia solution: 80 cc concentrated ammonia (sp. gr. 0.880) in 500 cc.

Hydrochloric acid: 60 cc concentrated hydrochloric acid in 500 cc.

Methyl orange solution:

Method of procedure.—An amount of material containing not more than 0.2 gram tartaric acid, not more than 0.3 gram alum, and not more than 0.3 gram calcium superphosphate is accurately weighed and placed in a dry flask. To this, 5 cc. of citric acid and 10 cc. of molybdate solution are added and allowed to react with the substance for 10 or 15 minutes (with an occasional shake). Next, 5 cc. of magnesium sulphate solution are added and 15 cc. of ammonia solution stirred in. After a few minutes (not more than an hour) the solution is filtered through a dry filter, a slight turbidity of the filtrate being disregarded. To 20 cc. of the filtrate are then added a few drops of methyl orange and hydrochloric acid, from a burette, till the pink color appears (2 or 3 drops too much or too little are of no consequence). Finally, 10 cc. more of the molybdate solution are added to the pink solution, which now becomes colorless or pale yellow; and water is added to make up the volume to 50 cc. This solution, after filtering if necessary, is polarized in a 20-cm. tube.

The amount of tartaric acid in grams (y) in the weight of substance originally taken is given by the following formula, in which x is the rotation in minutes:

$$y = 0.001086x + 0.001601\sqrt{x}.$$

But if the rotation is not less than 40', the simpler formula,

$$y = 0.0075 + 0.001168x,$$

may be employed.

The following table gives the tartaric acid in grams for every 10 minutes rotation:

Rotation in minutes.	Grams tartaric acid.	Rotation in minutes.	Grams tartaric acid.
10.....	0.016	90.....	0.1130
20.....	0.029	100.....	0.1246
30.....	0.0415	110.....	0.1365
40.....	0.0535	120.....	0.1479
50.....	0.0657	130.....	0.1595
60.....	0.0776	140.....	0.1710
70.....	0.0895	150.....	0.1825
80.....	0.1013		

It may be mentioned that the temperature has practically no influence on the readings.—*Edgar B. Kenrick, University of Toronto; Frank B. Kenrick, University of Manitoba.*

Page 106, 12, (b).—Has anyone ascertained how accurately a mixed precipitate of ferric and aluminum phosphates can be calculated into its components from its gross weight and ($\text{Al PO}_4=122$; $\text{FePO}_4=151$; $\text{P}_2\text{O}_5=142$) total phosphoric acid?

Let x and $y=\text{Al PO}_4$ and Fe PO_4 , respectively; then $x+y=a$;

$$\text{and } \frac{142x}{244} + \frac{142y}{302} = b.$$

Page 107, 12, (d).—Would you not usually recommend the indirect calculation of K from the weight of mixed chlorids and total chlorin as being much less laborious than the precipitate of $\text{K}_2\text{Pt Cl}_5$?—*A. McGill.*

FOOD PRESERVATIVES.

Page 108, 3.—It would be well to note that chloroform is much more convenient than ether for these extractions.

A more convenient way to apply the ferric chlorid test is to use a portion of the chloroform solution without evaporation. Shake this portion in a small test tube with about 1 cc of water, and add a very small drop of weak ferric chlorid solution; the violet color will be observed in the aqueous layer above the chloroform if salicylic acid is present. Successive small drops of ferric chlorid should be added until the deepest color is obtained or until it is evident that salicylic acid is not present. An excess of ferric chlorid must be avoided, as it tends to destroy the color; but, on the other hand, if the first addition of ferric chlorid does not produce the reaction, enough must be added to make sure that the color can not be obtained.

Page 109, 4.—The oxidation method devised by the writer (see Bulletin No. 59, U. S. Department of Agriculture, Division of Chemistry, page 60; or Leffmann's Select Methods in Food Analysis, 1901, page 98), based upon the oxidation of benzoic into salicylic acid (Hanriot, C. R. 102, page 1250), has been in use in this laboratory for nearly three years with good results, and I think it worthy a place in our methods. Recently Mr. J. O. LeBach, food chemist of this station, has simplified the manipulation by doing away with the cooling in ice water and performing the oxidation in a test tube instead of a dish. Mr. LeBach describes his method of making the test as follows:

Transfer about 0.05 gram to 0.1 gram of the dry residue from the chloroform or ether extract of the suspected sample to a test tube holding about 50 cc; add from 5 cc to 8 cc of concentrated H_2SO_4 , shake until the mass is dissolved, then add from 0.5 to 0.8 gram of barium peroxid, a little at a time, shaking and cooling in water after each addition of the peroxid. After all the barium peroxid has been added a permanent froth should have been formed on the sulphuric acid. Let stand from 20 to 30 minutes, then fill the test tube three-fourths full of water, shake and cool rapidly to the temperature of the room. Filter off the barium sulphate and extract the filtrate with chloroform or ether. Draw off the chloroform and test it for salicylic acid in the usual way with dilute ferric chlorid.

It is important that during the oxidation of benzoic acid to salicylic acid with barium peroxid the temperature of the solution should not go higher than ordinary temperature; the cooler the better.

I have been able to oxidize benzoic acid to salicylic acid with commercial hydrogen peroxid, but have not found the method as easy to control as the one given above. Ammonium persulphate works as well in most cases, but sometimes fails to act properly. With barium peroxid I have never had a failure.

We find that saccharin, when oxidized in this manner, gives the salicylic acid reaction. It is therefore necessary, before applying this test, to determine by the taste whether saccharin is present. Of course it is necessary also first to prove the absence of salicylic acid.—*Alfred M. Peter.*

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U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 66.

H. W. WILEY, Chief of Bureau.

FRUITS AND FRUIT PRODUCTS:

CHEMICAL AND MICROSCOPICAL
EXAMINATION.

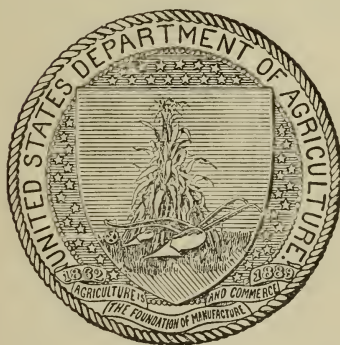
PREPARED UNDER THE DIRECTION OF

W. D. BIGELOW,

CHIEF OF FOOD LABORATORY,

BY

L. S. MUNSON, L. M. TOLMAN, AND BURTON J. HOWARD.



WASHINGTON:
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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,

Washington, D. C., January 25, 1902.

SIR: I have the honor to transmit herewith the manuscript of a report on our investigations of fruit and fruit products, with the request that it be published as Bulletin No. 66 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

INTRODUCTION.

Questions affecting the purity of staple foods are of vital interest to practically our whole population. This interest is increased, from the moral and commercial standpoints, with foods produced and manufactured in this country.

During recent years the cultivation of fruit and the manufacture of fruit products have reached such proportions that information is often sought regarding the true character of the preparations on the market.

The adulterants mentioned in the following pages are to be criticised on the ground of deception rather than because of their being prejudicial to health. At the same time, salicylic acid and saccharin are regarded with disfavor by the majority of disinterested investigators, it is conceded that benzoic acid should be subjected to further study before its use be unrestricted, and it sometimes happens that colors are employed that are not altogether lacking in injurious properties. There is a demand for further study of the effects of preservatives in general on the health of the consumer.

As stated above, however, the great majority of samples reported in the following pages as adulterated are probably not injurious articles, and their sale under proper labels is not open to objection. Correct labeling, however, is essential to the welfare of reliable manufacturers as well as that of consumers.

Many large manufacturers find it advantageous to add a commercial preservative to hold fruit in a partially prepared condition and finish it up at their convenience; to add apple juice, not as a "make weight" alone, but to insure a good, firm jelly; and to employ artificial colors to compensate for dilution with apple juice and to prevent fading on the grocers' shelves.

When such practices as the above are not indicated on the label, jellies made of a given fruit and sugar alone are subjected to unfair competition. The two articles may be equally wholesome, but there are many who prefer the latter and are willing to pay for the increased cost of manufacture.

There are numerous small establishments engaged in the preparation of "home-made" jellies and jams that are really true to name, and the number of private families that add something to their income in the same way is very great. Their welfare requires correct labeling of imitation products.

Moreover, the consumer has a right to expect that food products shall be true to label in every respect. There should be no misrepresentation regarding quality of product, variety of fruit employed,

place of production, or name of manufacturer. It is a common practice, for instance, to label all pears as Bartletts. This works injustice to both producers and consumers. Many canners place their own labels only on their best brands and pack inferior goods under the names of fictitious firms. This practice is less objectionable when the goods can be readily traced to the manufacturer, but it often happens that letters addressed to such fictitious firms are returned to the sender. The practice becomes most reprehensible when such inferior articles are marked "first quality."

Another form of mislabeling often resorted to is the branding of fruits of one locality with the name of a State or district which has attained an enviable reputation for the production of certain fruit. In this connection may be noted an injunction recently obtained in the Baltimore courts restraining certain packers from labeling their wares as California products.

For the purpose of comparison numerous analyses of fruits have been compiled, and fresh fruits, and jellies and jams prepared in the laboratory, have been examined.

The samples of fruit products examined were taken at random, no attempt being made to secure either high or low grade goods. On the contrary, attempts were made to secure a set of samples that would be thoroughly representative of the fruit products on the market. This matter is attended with great difficulty. The various manufacturers do not send their high-grade goods to the same markets. It often happens that a given firm which makes several brands of goods only finds sale for its lowest grade in some cities, where some manufacturers place only their best goods. The same conditions apply to different stores in the same city. It is quite possible that we have secured samples of high-grade goods of some manufacturers and overlooked stores which handled their cheap products, or vice versa.

As stated above, however, the samples were collected in such a manner as to preclude these conditions as far as possible. The cities of Washington, New York, Philadelphia, and New Orleans were visited by representatives of the Bureau and a list of stores selected where it was thought all grades of goods could be found. Samples were secured of all jellies, jams, and similar preparations on sale at these stores. A few additional samples were received from other sources.

All of the work in connection with this bulletin was accomplished by those whose names appear on the title-page, except the nitrogen determinations, which were made by Mr. T. C. Trescott.

As indicated above, the primary purpose of the work was to determine the character of the fruit products on the market. At the same time, the analyses given are of scientific value in extending our knowledge of the composition of fruits.

W. D. BIGELOW,
Chief of Food Laboratory

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FRUITS AND FRUIT PRODUCTS.

PART I.—CHEMICAL EXAMINATION OF FRUITS AND FRUIT PRODUCTS.

By L. S. MUNSON and L. M. TOLMAN.

METHODS OF ANALYSIS.

GENERAL DISCUSSION.

In making an examination of fruits or fruit products much depends upon the object of the analysis. Hence the preparation of the samples, the portions used for analysis, and the determinations to be made, must be left largely to the judgment of the analyst. In the examination of fresh fruits the determinations of pressed pulp and juice, the amount of waste material, and the relation between flesh and pit are often of considerable value. The examination should be so complete as to leave no doubt regarding the interpretation of the data obtained. It should be borne in mind that the more exhaustive the examination the greater is its scientific value and the broader is the view it gives of the nature of the product.

PRELIMINARY EXAMINATION.

Much valuable information may be obtained by a careful inspection of the samples under examination. The general appearance of fresh fruits, their size, color, and taste, are valuable in determining the quality. In the case of jellies and other fruit products preserved in glass this examination is of great importance. The label should be preserved and the general appearance, taste, odor, color, and consistency should be carefully noted, as they are valuable indications of the quality of the goods. The capacity of the receptacle should be noted by either measuring its volume or weighing the contents. In jellies containing starch there is a turbidity and often appreciable amounts of insoluble material, while in its absence the solution is perfectly clear. By this inspection low-grade fruits may be detected in jams, and an examination of the seeds may show the presence of foreign fruits. In the case of products put up in tins, any bulging of the can or escape of gas on opening, showing incomplete sterilization, should be care-

fully noted. The interior of the can will often show blackening or corrosion due to the attack of the metal by the fruit juices. This is especially true of goods that have undergone some fermentation or decomposition in the can.

PREPARATION OF SAMPLES.

FRESH FRUITS.

Pulp the whole, well cleaned, edible portion of the fruit in a large mortar, or by means of a food chopper, and mix thoroughly. With small fruits the entire fruit is employed, no effort being made to separate the seeds; with the apple, pear, and like fruits, the core is first removed, while with stone fruits the pits are separated. If desired the percentage of nonedible material may be determined in a weighed portion. Samples must be kept in well-stoppered bottles and in a cool place. Owing to the fact that fermentation begins in a very short time it is necessary to make the determinations of total and volatile acidity and sugars and the polarizations at once. Portions for polarizations and reducing sugars may be weighed and an excess of lead subacetate added. The samples may then be kept several days without danger of undergoing fermentation. The remainder of the sample may be preserved with about one-half cubic centimeter of formalin without danger of affecting the other determinations. It must be remembered that the sample so preserved can not be used for the determination of reducing sugars.

JUICES AND JELLIES.

Prepare the juices by pressing in a jelly bag a portion of the pulped fruit described under fresh fruits. Unless a clear liquid is obtained either filter through a muslin filter or decant the clear portion after allowing the liquid to settle for about an hour. Juices should be separated as completely as possible from the pulp in order to obtain a fair sample, as it is known that the first and the last pressings vary materially in composition. Colby,^a of the California Experiment Station, finds it advantageous in case of such fruits as apricots and prunes, which are difficult to press in the fresh state, to warm them sufficiently to soften, but not to drive off any water. After this treatment the juice is much more easily separated. He considers the determination of the relative amounts of juice and pressed pulp of value in comparing the juiciness of such fruits as oranges, lemons, etc., and that the determination may to some extent serve as a guide for canners. By pressed pulp is meant the material left after drawing off the juice, consisting largely of fiber and water, some of the ash, and most of the insoluble nitrogen compounds.

^a Communication by letter.

In the case of jellies, thoroughly mix to insure uniformity in sampling. Place 60 grams in a 300 cc flask, dissolve in water by means of frequent shaking, make up to the mark with water, and use aliquot portions for the various determinations. With jellies that contain starch or other insoluble material thoroughly mix before taking aliquot portions for the various determinations.

JAMS, MARMALADES, PRESERVES, AND CANNED FRUITS.

Thoroughly pulp the entire contents of the jar or can as directed under fresh fruits. With this class of fruit products no effort need be made to remove any of the nonedible portions except the pits. Pears and similar fruits if put up as whole fruit are prepared for analysis without the removal of the cores.

In the examination of canned fruits it is often sufficient, when the analysis is for the sole purpose of detecting adulterants, merely to examine the sirups in which the fruits are preserved, as the sirup will contain the added ingredients, such as glucose and preservatives. In some products, however, such as Maraschino cherries, the fruit itself is colored with a coal-tar dye which is insoluble in the sirup, and it is necessary to pulp these in order to extract the color for identification. In such a case the liquor may be separated and the relative amounts of liquid and solid portions determined, as this may be of value in showing the presence of an excessive amount of water. The examination of the sirup need not be as complete as when the whole fruit is taken.

The price of the goods, as a rule, depends largely on the amount of sugar in the sirup.

DETERMINATION OF TOTAL SOLIDS.^a

The determination of total solids was the subject of considerable work before the method of drying at 100° C. was finally adopted. The drying of the samples in vacuo was not considered practicable, as few laboratories are equipped to use this method, and the large bulk of work also prohibited it. There can be no doubt, however, that this is the most accurate method with samples containing large amounts of invert

^a MCGILL'S WATER OVEN.—A. McGill has devised a forced-draft water oven for drying at temperatures between 60° and 90° C. The oven is heated by means of ordinary gas burners, and the temperature is controlled by introducing at the bottom of the oven a blast of air from a blower that is run by a small water motor. Before discharging into the oven the air tube (which is about 1 inch in diameter) enters the water chamber and is coiled a number of times in order to sufficiently warm the air before it enters the oven. The exit end of the air tube is covered with a concavo-convex disk in order to distribute the blast and to prevent harmful currents. By regulating the burners and the flow of air a fairly constant temperature can be obtained. The bottom of the oven is curved instead of flat, to prevent bumping when the water is boiling, and a perforated plate serves as a false bottom.

sugar, as it has been shown^a that levulose is dehydrated at 100° C. Of the other two methods employed the one depending upon the calculation of solids from the specific gravity of the 20 per cent solution gives results more nearly concordant with drying in vacuo than by drying at 100° C., the results being from 1 to 1.50 per cent higher than those obtained with the latter method. With pure jellies the specific gravity method gives very satisfactory results, but with glucose products containing large amounts of dextrin and with jellies containing soluble starch this method is not so reliable. For the purpose of obtaining comparative results with all classes of fruits and fruit products and with the different grades of goods the method of drying at 100° C. for from twenty to twenty-four hours is considered satisfactory. The method is an empirical one and all details must be followed closely in order to obtain comparative results. When neither sand nor asbestos was used as an absorbent the results were unsatisfactory and from 2 to 3 per cent too high.

IN JUICES AND JELLIES.^b

By direct determination.—Measure 25 cc of a 20 per cent solution of jelly, or weigh 25 grams of juice into a large flat-bottomed dish which contains about 4 or 5 grams of freshly ignited asbestos to absorb it; dry for from twenty to twenty-four hours^c in a water-jacketed oven. If care is taken, measuring will be found to be as accurate as weighing, but the pipette must be graduated to deliver 25 cc of a 20 per cent sugar solution. In the case of jellies that contain starch or other insoluble matter solids may be determined as directed under fresh fruits, jams, marmalades, etc.

By calculation from specific gravity.—Determine the specific gravity of the solution of jelly, or of the juice, by means of a Westphal balance, pycnometer, or specific-gravity spindle, and calculate the solids from the table given in bulletin 65.

IN FRESH FRUITS, JAMS, MARMALADES, PRESERVES, AND CANNED FRUITS.

Place a weighed quantity of about 20 grams of pulped fresh fruit, or such an amount of fruit product as will give not more than 3 or 4 grams of dried material, in a large flat-bottomed dish containing from 4 to 5 grams of ignited asbestos; add a few cubic centimeters of water, mix thoroughly, and dry as under "Direct determination" in "Juices and jellies."

^aCarr and Sanborn, U. S. Dept. of Agric., Div. of Chem. Bull. 47, p. 134.

^bMcGill by letter recommends the use of the Macfarlane tube described in the Analyst, 1893, 18, 73, for the determination of total solids in substances containing large amounts of sugar.

^cWiley, Principles and Practice of Agricultural Analysis, vol. 3, p. 579, recommends drying first at a low temperature and completing the operation at 100° C. or a little higher.

DETERMINATION OF INSOLUBLE SOLIDS.

KREMLA'S METHOD.^a

Fifty grams of the sample are weighed, transferred to a mortar, and thoroughly triturated. The mass is then transferred to a muslin filter and washed with warm water, care being taken at each addition of water to stir the pulp thoroughly. Collect the filtrate in a 500 cc flask, cool, and make up to volume. Usually this amount is sufficient to completely remove all soluble material; in some cases, however, it may be necessary to increase the washings to 1,000 cc. The insoluble residue is transferred to a tared dish, the excess of water is evaporated, and the residue dried in a water oven at 100° C. for four hours before weighing. The filtrate may be slightly turbid, showing that some of the insoluble matter has passed through the filter, but this amount will be inappreciable. Kremla used cold water and coarse filter paper, and the method has been modified in this work in these respects,

GERMAN OFFICIAL METHOD.^b

Transfer a weighed portion of the fruit product to a graduated flask, add water, shake thoroughly, and make up to volume.^c Allow this to settle and either filter or decant off the supernatant liquid. Take an aliquot portion for the determination of soluble solids. Total solids less the soluble solids equals the insoluble solids.

This method may be employed to advantage with such fruits as cherries and small fruits, with which even the filtration through muslin is made with difficulty, but care must be taken that the fruit is thoroughly macerated to insure complete solution of the soluble matter.

DETERMINATION OF ASH.

Evaporate to dryness in a large platinum dish, 50 cc of a 20 per cent solution of jelly (see p. 11, under Juices and jellies), 25 grams of juice or fresh fruit, or 10 grams of jam, marmalade, preserve, or canned fruit; then thoroughly char at as low a heat as possible, extract with water, filter, and wash. Return the filter paper and insoluble material to the dish and thoroughly ignite; add the soluble portion and evaporate the whole to dryness after adding a few cubic centimeters of a solution of ammonium carbonate; then heat for a moment to very low redness; cool in a desiccator and weigh. The weighing must be made as quickly as possible, as the ash absorbs moisture very rapidly.

^a Ztschr. Nahr. Hyg. Waar., 1892, 6, 483.

^b Ver. Nahr. u. Genussm. f. Deutsche Reich., 2, 105.

^c McGill by letter recommends the use of a mechanical shaker to obtain complete solution of the soluble material.

EXAMINATION OF ASH.

ALKALINITY.

Into the platinum dish containing the ash run an excess of fifth-normal nitric acid and add a few drops of methyl orange. Carefully rub up the ash with a rubber-tipped stirring rod and titrate the excess of acid with decinormal potassium hydroxid. Calculate the alkalinity as per cent of potassium carbonate in the original substance. One cubic centimeter of decinormal acid equals 0.00691 gram of potassium carbonate.

SULPHATES AND CHLORIDS.

Wash the ash into a 50 cc flask and make up to the mark with water. In 25 cc of this solution determine the sulphates by precipitation with barium chlorid. The weight of barium sulphate times 0.7478 gives the weight of sulphates calculated as potassium sulphate.

In the other portion of the solution determine the chlorids by the Volhard^a method for chlorin. The nitric acid added before making the titration will, if it contain enough nitrous oxid, completely destroy the red color of the methyl orange and leave a clear solution for the titration. Calculate the chlorin as sodium chlorid.

The determination of sulphates and chlorids in the amount of material above named is possible only with fruit products containing glucose. The ash from this amount of pure fruit juice or pure fruit products will contain only traces of sulphates and chlorids, while that from glucose products will contain determinable amounts of either sulphates or chlorids or both, depending upon the processes of manufacture of the glucose. If a complete ash analysis is to be made, the directions given in the official methods^b may be followed.

DETERMINATION OF TOTAL ACIDS.

Ten grams of the juice, fresh fruit, or fruit product, or 25 cc. of the solution of jelly are diluted with boiling distilled water to about 250 cc. In the case of fruit pulp it takes some time to dissolve all the acidity from the fruit cells, and it is well to boil for a minute or so in order to aid the solution. A smaller volume may be employed if the product is not highly colored. Add phenolphthalein and titrate the acid with decinormal potassium hydroxid. Frequently with highly colored products it is impossible to determine with accuracy the end reaction in case phenolphthalein is used. In such cases delicate litmus paper may be used to advantage, but the product must not be highly

^a Ann. der Chem., 1877, **190**, 1; Sutton's Volumetric Analysis, 8th edition, p. 155.

^b U. S. Dept. of Agr., Div. of Chem. Bul. 46, revised, p. 77.

diluted. Calculate the results as sulphuric acid (H_2SO_4). One cubic centimeter of decinormal potassium hydroxid equals 0.0049 gram of sulphuric acid.

Sulphuric acid was adopted as the term for the expression of acidity of fruit products because of its convenience in allowing comparison. If a single organic acid were the common and dominant acid of all the fruits that acid would serve the purpose better than the one chosen. But such is not the case. Citric, tartaric, and malic acids are each dominant in certain fruits, and the acidity of at least a large number of the fruits is due to mixtures of two or more of the organic acids. Besides, a part of the acidity may be due to the presence of acid salts, so that an attempt to express the total acidity in terms of a single organic acid characteristic of the fruit would meet with obvious difficulties. Sulphuric acid has already been suggested and adopted by a number of laboratories for similar work, and it is accepted here as offering the most satisfactory basis for the expression of acidity in fruits and fruit products.

DETERMINATION OF VOLATILE ACIDS.

The determination of volatile acids in fruit products may be desirable in cases where fermentation or the use of decayed fruit is suspected. Dissolve 25 grams of substance in water, dilute to 50 cc and distil in a current of steam until about 200 cc have passed over. Titrate the distillate with decinormal potassium hydroxid and express the results as acetic acid. Each cubic centimeter of decinormal alkali is equivalent to 0.0060 gram of acetic acid.

It should not require more than a few tenths of a cubic centimeter of decinormal alkali to neutralize the volatile acid obtained from this amount of fresh fruit.

DETERMINATION OF FREE MINERAL ACIDS.

A. S. Mitchell^a has called attention to the presence of free sulphuric and phosphoric acids in jellies, added to gelatinize the skins, cores, and other waste material of the fruit. Phosphoric acid was found in several jellies (see Table 31), one of which (659) had an acid ash and another (660) an ash that was practically neutral.

Sulphuric acid may be estimated by the Hehner method used in determining free mineral acids in vinegar. To a weighed quantity of the jelly add an excess of decinormal alkali, evaporate to dryness, ash, and titrate the ash with decinormal acid. The difference between the number of cubic centimeters of alkali added in the first place and the number of cubic centimeters of acid needed to titrate the ash represents the equivalent of the amount of free mineral acid present. Phosphoric acid can be estimated in the ash.

^a Communication by letter.

DETERMINATION OF TARTARIC, CITRIC, AND MALIC ACIDS.^a

Use the filtrate from the alcohol precipitate in the determination of the organic acids. After evaporating the alcohol and taking up the acids with water, add lead subacetate until the solution is alkaline, then filter and wash the precipitate until only a slight amount of lead remains in the washings. Wash the precipitate off the filter paper into a beaker with hot water, precipitate the lead with hydrogen sulphid and filter the lead sulphid while hot, washing with hot water. Evaporate the filtrate which contains the free organic acids to about 50 cc and neutralize exactly with potassium hydroxid, using phenolphthalein as indicator. Add an excess of strong solution of neutral calcium acetate with constant stirring, and allow to stand from six to twelve hours. Throw the precipitate of calcium tartrate on a filter paper and wash until filtrate and washings make exactly 100 cc; ignite the filter paper and precipitate, determine the lime by titration, and calculate the tartaric acid therefrom. A correction of 0.0286 gram of tartaric acid which is held in solution in the 100 cc of washings as calcium tartrate must be added. Evaporate the filtrate to about 20 cc, and if a precipitate of calcium citrate is formed filter it hot, wash with hot water, ignite, and titrate the lime. From this calculate the citric acid. Again evaporate the filtrate to about 20 cc and add 3 volumes of 96 per cent alcohol by volume, which will throw down the calcium salt of tartaric acid held in solution, the rest of the citrate, and the malate and succinate. Filter this, ignite, titrate, and after subtracting the tartaric acid present, calculate the rest as malic acid, since the amount of citric and succinic acid present is very small.

DETERMINATION OF TARTARIC ACID.^b

To 100 cc of the fruit juice add 2 cc of glacial acetic acid, 2 or 3 drops of a 20 per cent solution of potassium acetate, and 15 grams of pure, finely powdered potassium chlorid. Dissolve this by shaking and then add 20 cc of 96 per cent alcohol. Stir vigorously for one minute, rubbing the walls of the beaker with the glass stirring rod to start the crystallization of the potassium bitartrate, and allow to stand for fifteen hours at room temperature. Filter and transfer the precipitate to a Gooch crucible with a thin asbestos felt and wash with a mixture of 15 grams of potassium chlorid 20 cc of alcohol and 100 cc of water, using the vacuum pump to aid filtration. The beaker is rinsed three times with a few cubic centimeters of this solution, and the precipitate is also washed with it, but in such a way that not more than 20 cc in all of the wash solution are used. The precipitate and asbestos filter are washed into the beaker with water and heated to boiling.

^a A modification of Schmidt and Hiepe's method. U. S. Dept. of Agric., Div. of Chem. Bul. 46, revised, p. 67. Ztsch. anal. Chem., 1882, **21**, 534-541.

^b Halenke and Möslinger, Ztschr. anal. Chem., 1895, **34**, 283.

While still hot the solution is titrated with decinormal alkali, using phenolphthalein as indicator. To the number of cubic centimeters of alkali used must be added 1.5 cc for the potassium bitartrate remaining dissolved in the solution. One cubic centimeter of decinormal alkali equals 0.0150 gram of tartaric acid.

DETERMINATION OF CITRIC ACID.^a

Fifty cc of the fruit solution are evaporated to sirupy consistency on the water bath. To this residue add 95 per cent alcohol until no further precipitation is formed, pouring very slowly at first and stirring constantly. From 70 cc to 80 cc are generally sufficient. Filter and wash the residue with alcohol of approximately 95 per cent. Evaporate the filtrate to eliminate alcohol, take up the residue with a little water, and transfer to a graduated cylinder, making up to 10 cc. To 5 cc of this solution add half a cubic centimeter of glacial acetic acid and then drop by drop a saturated solution of lead acetate. The presence of citric acid is shown by the appearance of a precipitate which disappears on heating and reappears on cooling. In order to separate the citric acid from other acids, heat to boiling, filter and wash with boiling water, then allow to cool, and the precipitate of lead citrate will reform. This lead precipitate may be filtered, washed with dilute alcohol, dried, weighed, and the citric acid calculated.

It is necessary that there shall be no tartaric acid present. If the tartaric acid has been estimated this error can be avoided by adding enough decinormal potash to exactly neutralize the amount of tartaric acid present before adding the alcohol.

DETERMINATION OF NITROGEN.

Use 5 grams of jelly or other fruit product or 10 grams of juice or fresh fruit for the determination of nitrogen according to either the Gunning or the Kjeldahl method. Express results as protein (nitrogen multiplied by 6.25).

DETERMINATION OF ALCOHOL.

Transfer 50 grams of the original material to an Erlenmeyer flask of from 250 to 300 cc capacity and increase the volume of the liquid to 150 cc. Attach to a vertical condenser by means of a bent tube and distill 100 cc. Determine the specific gravity of the distillate and calculate the percentage of alcohol from a suitable table.^b Unless the specific gravity is taken at 15.6° the temperature correction may be made according to Table III, Bulletin 59, p. 95. Determinations should be made, however, at as nearly the standard temperature as practicable.

^aMöslinger, *Ztschr. Unter. Nahr. u. Genuss.* 1899, 2, 93.

^bU. S. Dept. of Agr., *Div. of Chem. Bul.* 59, p. 65.

Where occasional determinations of alcohol are made it is found convenient to use an alembic saleron. This apparatus is made of copper and can be readily taken apart and placed in a small box. No rubber connections are necessary, so that the setting up requires only a few minutes.

POLARIZATION.

Dissolve half the normal weight of jelly or other fruit product, or the normal weight of juices or fresh fruits, in a sufficient quantity of water in a 100 cc sugar flask, add an excess of lead subacetate^a (from 5 to 10 cc), make up to 100 cc, filter, and polarize in a 200 mm tube, observing the temperature of the solution. Invert 50 cc of this solution, using 5 cc of hydrochloric acid by heating to 68° in fifteen minutes. Polarize in a 220 mm tube at the same temperature as was employed in making the direct reading.

On account of the large amounts of invert sugar usually found in these products, it is necessary that the direct and invert readings should be made at the same temperature.

DETERMINATION OF CANE SUGAR.

BY CALCULATION FROM CLERGET'S FORMULA.

The cane-sugar results given in this bulletin were calculated from the direct and the invert readings according to Clerget's formula:

$$S = \frac{(a-b) 100}{144 - \frac{t}{2}}$$

This method is as accurate as any for the examination of glucose goods. In the absence of glucose much greater accuracy may be secured by using a modified formula, which will correct for the change in optical power of the invert sugar present at the time of the direct polarization, caused by the addition of hydrochloric acid used in making the inversion. This formula, which is based on the effect of hydrochloric acid on solutions of invert sugar containing citric acid and on solutions containing no acid at all, is as follows:

$$S = \frac{[A - (B - .062B)] 100}{141.79 - \frac{t}{2}}$$

S = cane sugar.

A = direct reading at t°.

B = invert reading at t°.

t = temperature.

^a Prepared by boiling for half an hour 430 grams of normal lead acetate, 130 grams of litharge, and 1,000 cc of water. The mixture is allowed to cool and settle, when the supernatant liquid is diluted to 1.25 specific gravity with recently boiled water.

This formula is that proposed by Tolman,^a and is a modification of the Clerget-Herzfeld formula. The factor .062B is the increased reading to the left, due to the presence of 10 cc of hydrochloric acid (specific gravity 1.20) in 110 cc of invert sugar solution.

By the use of this formula it will be found that in the examinations of many fruits and fruit products the apparent change in polarization of a degree or two after inversion is not due to the presence of cane sugar, but to the change brought about by the addition of the acid used in inversion. It must be borne in mind that this formula is not applicable in the presence of glucose, but is of special value in the accurate determination of small amounts of cane sugar in fruits and fruit products.

Samples containing glucose may give results at least 1 per cent too high, which will give an invert reading too low, owing to the action of hydrochloric acid upon the maltose and dextrin. As will be seen by reference to the tables, many of the glucose products show small amounts of cane sugar (0.5 to 1.5 per cent), while, as a matter of fact, this is an error due to the cause stated.

BY COPPER REDUCTION.

When only a small amount of cane sugar is present, it is best determined by calculation from the increase in reducing sugars after inversion. For this purpose treat double the amount of fruit or fruit product named under "Reducing sugars" with lead subacetate, and after making up to volume and filtering, invert 50 cc in a 100 cc flask with 5 cc of hydrochloric acid. After inversion neutralize the acid with sodium hydroxid, precipitate the excess of lead with sodium sulphate, and dilute with water to 100 cc. Filter and dilute so that the solution does not contain more than 1 per cent of reducing sugar. The per cent of increase in reducing sugar after inversion multiplied by 0.95 equals the per cent of cane sugar.

DETERMINATION OF REDUCING SUGARS.

Treat 5 grams of jelly (25 cc of a 20 per cent solution may be employed) or other fruit product, or 25 grams of juice or fresh fruit, with lead subacetate in excess (2 to 5 cc); make up to 100 cc and filter. Transfer from 25 to 50 cc—depending upon the percentage of reducing sugar present—to a 100 cc flask and add a saturated solution of sodium sulphate in sufficient amount to precipitate the excess of lead; complete the volume to 100 cc and use the filtered solution for the determination of reducing sugars. The approximate amount of reducing

^a Jour. Amer. Chem. Soc., 1902, 24, 515.

sugar present may be readily ascertained from the polarizations and from the per cent of solids. Use Allihn's method for the determination^a and express results as dextrose, making the calculation from Allihn's tables.

Owing to the varied nature of the reducing sugars found in fruits and fruit products, and to the fact that no table has been constructed that will in all cases apply, it was thought best to express all results as dextrose, although it is well known that with pure invert sugar, as well as with glucose products, a material error will arise. In no case do we find that the reducing sugar is pure dextrose. With pure fruits and fruit juices the reducing sugars are made up of practically equal portions of dextrose and levulose. The same holds true with pure-fruit products, in which the invert sugar of the fruit is greatly increased by the inversion of the cane sugar by the organic acids present. With fruit products containing glucose, on the other hand, the problem is more complicated. Here is found not only invert sugar from the fruit used, but also dextrose and maltose, which are normal constituents of glucose sirups. If cane sugar were also used part may become inverted and thus add to the invert sugar. Since the reducing power of dextrose, levulose, and maltose vary widely, the expression of reducing sugars as dextrose in case of mixtures of the three sugars gives results far from the truth. The presence of cane sugar is also a disturbing factor, as it materially influences the power of reduction, depending upon the amount present. The quantity of invert sugar, where no other sugar is present, can be determined from reduced copper by reference to the table of Meissl and Wein.^b Wein^c has constructed tables from results obtained by Meissl for the calculation of reducing sugars in the presence of cane sugar from the amount of copper reduced, but these tables hold good only with pure fruits or pure-fruit products, and are not reliable when dealing with glucose products.

DETERMINATION OF DEXTRIN.

Dissolve 10 grams of the sample^d in a 100 cc flask; add 20 mg of potassium fluorid and then about one-quarter of a cake of compressed yeast.^e Allow the fermentation to proceed below 25° C. for two or three hours to prevent excessive foaming, and then place in an incubator at a temperature of from 27° to 30° C. for five days. At the end of that

^a U. S. Dept. of Agr., Div. of Chem. Bul. 46, revised, p. 35.

^b Wiley, Principles and Practice of Agricultural Analysis, Vol. III, pp. 159-160.

^c Tabellen zur Quantitativen Bestimmung der Zuckerarten.

^d In the case of jellies 50 cc of a 20 per cent solution prepared as directed (p. 11) may be used.

^e Bigelow and McElroy, Jour. Am. Chem. Soc., 1893, 15, 668,

time clarify with lead subacetate and alumina cream,^a make up to 100 cc, filter, and polarize in a 200 mm tube. A pure fruit jelly will show a rotation of not more than a few tenths of a degree either to the right or to the left. If a Schmidt and Haensch polariscope be used and a 10 per cent solution is polarized in a 200 mm tube, the number of degrees read on the sugar scale of the instrument multiplied by 0.8755 will give the percentage of dextrin; or the following formula may be used:

$$\text{Percentage of dextrin} = \frac{C \times 1,000 \times V}{198 \times L \times W}$$

in which—

C = degrees of circular rotation.

V = volume in cubic centimeters of solution polarized.

L = length of tube in centimeters.

W = weight of sample in solution in grams.

DETERMINATION OF ALCOHOL PRECIPITATE.

Evaporate to 20 cc 100 cc of a 20 per cent solution of jelly, or 200 cc of the washings from the determination of insoluble solids; then add slowly and with constant stirring 200 cc of 95 per cent alcohol by volume, and allow the mixture to stand overnight. Filter and wash with 80 per cent alcohol by volume. Wash this precipitate off the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100° C. for several hours and weigh; then burn the organic matter and weigh the residue as ash, after treating with ammonium carbonate as directed under determinations of ash (p. 13). The loss in weight upon ignition is called alcohol precipitate.

The ash should be largely lime, and not more than 5 per cent of the total weight of the alcohol precipitate. If it is larger than this it is due to the presence of salts of the organic acids. Titrate the water-soluble portion of this ash with decinormal acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

The general appearance of the alcohol precipitate of fruit products is one of the best indications as to the presence of glucose. Upon the addition of alcohol to a pure fruit product a flocculent precipitate is formed with no turbidity, while in the presence of glucose a white turbidity appears at once upon adding the alcohol, and a thick, gummy precipitate forms. In fresh fruit juices there is often a marked turbidity, closely resembling the precipitate given by glucose, which is caused by the starchy matters present.

^a Wiley, Principles and Practice of Agricultural Analysis, Vol. III, p. 100, gives the following method for preparation of alumina cream: A solution of alum is treated with ammonium hydroxid and the precipitate washed until it is freed from ammonia. The hydroxid is then suspended in water in proportions necessary to produce a creamy liquid.

DETERMINATION OF TANNIN AND COLORING MATTER.

Tannin and coloring matter are estimated in fruits and fruit products by the permanganate or Neubauer-Loewenthal method, as used in wine analysis.^a The following reagents are used:

(1) A solution of potassium permanganate containing about $1\frac{1}{2}$ grams of the salt to the liter.

(2) A decinormal oxalic acid solution for determining the strength of the permanganate solution; 1 cc equals 0.004157 gram of tannin.

(3) An indigo-carmin solution to be used as an indicator and containing 6 grams of indigo carmin (this must be pure and free from indigo blue) and 50 cc of sulphuric acid to the liter. Sodium sulphindigotate may be used instead of indigo carmin. Dissolve 6 grams of the salt in 500 cc of water by aid of heat, cool, add 50 cc of sulphuric acid, make up the solution to 1 liter, and filter.

(4) Pure boneblack which has been treated with hydrochloric acid and washed with water until free from acid. This is kept covered with water.

The method of procedure is as follows:

A large porcelain evaporating dish is used for the titration. About 750 cc of water are placed in it and from 25 to 50 cc of fruit juice added, then 20 cc of the indigo solution, and the whole is well mixed. The permanganate solution is then run in very slowly, with constant stirring, and at the last added only a drop or two at a time, allowing the reaction considerable time. The end point is the golden yellow. To another portion boneblack is added and allowed to stand fifteen minutes. It is then filtered and carefully washed with water. This filtrate is treated in the same manner as the original portion, and the difference between the number of cubic centimeters of permanganate used the first time and that used the second time is the amount used in the oxidation of the tannin and coloring matter.

DETECTION OF PRESERVATIVES.

Dissolve about 25 grams of the sample in water, acidify with dilute sulphuric acid (1 to 3), and extract with ether. Remove the ether layer and allow to evaporate spontaneously. Take up the residue, which may contain salicylic acid, benzoic acid, and saccharin, with a few cubic centimeters of water, and apply the tests given below for these substances. If it is desired to make tests for other preservatives, consult Bulletin 65.

SALICYLIC ACID.

To a few drops of the extract add 1 or 2 drops of a 0.5 per cent solution of ferric chlorid. A purple coloration indicates the presence of salicylic acid.

^a Wiley, Principles and Practice of Agricultural Analysis, Vol. III, p. 593; U. S. Department of Agr., Bureau of Chem. Bul. 65, p. 86.

BENZOIC ACID.

Mohler's test.—Treat a second portion of the extract with 2 to 3 cc of strong sulphuric acid, and heat until white fumes appear. By this means benzoic acid is converted into sulphobenzoic acid. Add a few crystals of potassium nitrate, and continue the heating, with repeated additions of potassium nitrate until the solution is colorless or of a very light yellow color. This causes the formation of metadinitrobenzoic acid. When cool dilute with about 5 cc of water, neutralize with ammonia, and transfer to a test tube. Filter if not clear or if crystals of ammonium or potassium sulphate separate. Add to the filtrate a few drops of ammonium sulphid, care being taken that it does not mix but forms a layer on the top. The nitro compound becomes converted into the ammonium metadiamidobenzoate, which possesses a bright cherry-red color. The reaction takes place in a few seconds, and is readily seen at the plane of contact of the two liquids. This reaction is also given by saccharin, but benzoic acid may readily be separated from saccharin by distillation and the test applied to the extract from the distillate.

SACCHARIN.

Saccharin is indicated by the sweet taste of the ether extract. To confirm this test, add to the remaining portion of the extract 1 or 2 grams of sodium hydroxid, and heat in an oil bath at a temperature of 250° C. for from twenty to thirty minutes. The saccharin is converted into "Salicylic acid." After cooling, dissolve in water, acidify with dilute sulphuric acid, extract with ether, and test for salicylic acid as described under salicylic acid. If salicylic acid is present in the original material this test, of course, can not be applied, but reliance must in that case be placed in the sweet taste of the extract.

DETECTION OF FOREIGN COLORING MATTER.

The complete examination of dyes is too large a subject to take up here, and one will have to refer to such works as Schultz and Julius on Organic Coloring, Allen's Commercial Organic Analysis, and others that go into the subject in an exhaustive manner. The determination of the general nature of the dye can be made by the use of Rota's^a scheme, which is the simplest of the many different methods proposed and is quite satisfactory, although it requires a great deal of care and experience.

The detection of the color in a fruit product and its identification are rather difficult, since the color must be separated in a somewhat pure condition and then tested. Almost all the methods for separating

^a Chem. Ztg., 1898, **22**, pp. 437–442; Analyst, 1899, **24**, p. 41; U. S. Dept. of Agric., Bureau of Chem. Bul. 65, p. 114.

added color from the fruit will take up some of the natural color of the food as well.

As will be seen in Tables I and II, amyl alcohol extracts the coloring matter from many fruits, and these extracts may easily be mistaken for added colors.

Some of the highly colored fruit juices will dye wool, and the color will be permanent, but these will not be mistaken for coal-tar dyes if the double dyeing method is followed.

Mixtures of two or more dyes are often added to foods. This can sometimes be shown by a system of fractional dyeing where the dyes are taken up at different rates by the fabric. In examining mixtures of red, orange, and blue dyes, which are widely sold for coloring wine, the writers found that the woolen cloth took up the red much faster than the orange, and the blue was slowest; so that the first piece of cloth dyed was red, the second a lighter shade, the third greenish, and the fourth bluish.

In the examination of the coloring matter for the heavy metals, tin, lead, copper, and zinc will be found in the ash, and tests can be made for them by the methods used for heavy metals.

DETECTION OF COAL-TAR COLORING MATTERS BY DYEING WOOL.

Method of Sostegni and Carpentieri.^a—From 10 to 20 grams of the sample are dissolved in 100 cc of water, filtered if necessary, acidified with from 2 to 4 cc of a 10 per cent solution of hydrochloric acid, and a piece of woolen cloth which has been washed in a very dilute solution of boiling potassium hydroxid and then washed in water, immersed in it, and boiled for five to ten minutes. The cloth is removed, thoroughly washed in water, and boiled with very dilute hydrochloric acid solution. After washing out the acid the color is dissolved in a solution of ammonium hydroxid (1 to 50). With some of the dyes solution takes place quite readily, while with others it is necessary to boil some time. The wool is taken out, a slight excess of hydrochloric acid is added to the solution, another piece of wool is immersed and again boiled. With vegetable coloring matter this second dyeing gives practically no color, and there is no danger of mistaking a fruit color for one of coal-tar origin. It is absolutely necessary that the second dyeing should be made, as some of the coal-tar dyes will dye a dirty orange in the first acid bath which might be easily passed for vegetable color, but on treatment in alkaline bath and second acid bath becomes a bright pink.

Arata's method.^b—This method gives results comparable with those

^a Ztschr. anal. Chem., 1896, **35**, 397; U. S. Dept. Agric., Div. Chem. Bul. 46, revised, p. 68.

^b Ztschr. anal. Chem., 1889, **28**, 639.

of the first dyeing of the preceding method. It was recommended for detecting coal-tar colors in wine, and has been modified by A. L. Winton.^a

From 20 to 30 grams of the sample dissolved in 100 cc of water are boiled for ten minutes with 10 cc of a 10 per cent solution of potassium bisulphate and a piece of white wool or woollen cloth which has been previously heated to boiling in a very dilute solution of sodium hydroxid and thoroughly washed with water. After removal from the solution the wool is washed with boiling water and dried between filter papers. If the coloring matters are entirely from the fruit the wool will be either uncolored or will take on a faint pink or brown, which is changed to green or yellow by ammonia and not restored by washing.

In addition to this it is advisable in all cases to dissolve the coloring matter with ammonia as in the first method and dye again, since Arata's method gives practically the same results as the first dyeing in hydrochloric acid bath, and needs to be substantiated by the second dyeing.

Another advantage in the second dyeing is that if a large piece of woollen cloth is used in the first dyeing and a small piece in the second dyeing small amounts of coloring matter can be brought out much more decidedly in the second dyeing where practically all of the vegetable coloring matter has been excluded. The coloring matter can be identified to a certain extent by the schemes of Witt,^b Allen,^c Weingartner,^d Dommergue,^e Girard and Dupré,^f and Rota.^g The tests can be made directly on the dyed fabric or the dye can be dissolved out.^h To remove the color wash the wool with dilute tartaric acid and then with water and dry between filter paper. Saturate the wool with strong sulphuric acid and press out the color with a glass rod after from five to ten minutes and dilute to 10 cc with water.

Remove the wool, make solution alkaline with ammonia, and when cold extract with from 5 to 10 cc of amyl alcohol. Separate the amyl alcohol, evaporate it to dryness, and test the residue. Treat this residue with strong sulphuric acid.

Ponceau R, 2R, 3R, S, and 3S give yellow red to carmine red.

Ponceau S and tropaeolin O give yellow to orange yellow.

Biebrich scarlet gives a green; Bordeaux red and crocein scarlet give blue; tropaeolin OOO and solid red give violet.

^a Conn. Exp. Sta. Rept., 1899, Pt. II, p. 131.

^b Ztschr. anal. Chem., 1887, **26**, 100.

^c Com. Org. Anal., Vol. III, pt. 1, pp. 399-420.

^d Ztschr. anal. Chem., 1888, **27**, 232-249.

^e Ibid., 1890, **29**, 369-377.

^f Analyse des Matières Alimentaires, etc., 583-593.

^g Analyst, 1899, **24**, 41.

^h Ztschr. anal. Chem., 1889, **28**, 639; Borde, Anal. des Weines, p. 91; Winton, Conn. Expt. Sta. Rept., 1899, Pt. II, p. 131.

If the wool is well dyed most of these colors may be obtained on the fabric.

This gives only the reactions of a few of the more common colors. In order to carry the work farther the more complete works referred to will have to be used.

DETECTION OF COAL-TAR COLORS BY EXTRACTION WITH SOLVENTS.

In the Paris municipal laboratory^a the following scheme of extraction of coal-tar color is used:

The acid colors, sulpho-fuchsin, azo derivatives, and phthaleins are not precipitated by tannin and are insoluble or only slightly soluble in acetic ether or amyl alcohol.

The basic colors (fuchsin, safranin, etc.) are precipitated by tannin and readily soluble in acetic ether or amyl alcohol.

I. To 50 cc of wine add ammonium hydroxid in slightest excess; then add 15 cc of amyl alcohol, shake, and allow to stand.

1. If the alcohol be colored red or violet, decant, wash, filter, evaporate to dryness in presence of a piece of wool, and test the dyed wool with sulphuric acid.

2. If the alcohol be not colored, separate, and add acetic acid. If the alcohol becomes colored the presence of basic aniline color is indicated.

3. If the amyl alcohol is uncolored, both before and after the addition of acetic acid, no basic coal-tar color is present.

II. Add an excess of calcined magnesia and then a 20 per cent solution of mercuric acetate and bring to a boil. A coloration before or after addition of acetic acid indicates the presence of coal-tar dyes, particularly acid dyes.

III. Extract with acetic ether the solution made alkaline by barium hydroxid. This dissolves basic colors.

In any case the colors must be fixed on wool, as many of the fruit colors are extracted and will give reactions with sulphuric acid, which may be mistaken for coal-tar colors.

The extraction of fruit colors is shown in Table 1, prepared by Truchon and Martin-Claude,^b and Table 2, prepared by the writers. The fresh fruit juice was very slightly acidified by hydrochloric acid before extraction. In no case in the dyeing test was there any danger of mistaking the vegetable color for one of coal-tar origin where the double dyeing method was used.

^a Girard and Dupré, *Analyse des Matières Alimentaires*, etc., p. 167.

^b *Journ. pharm. chim.*, 1901, **13**, 174.

TABLE 1.—*Coloring matter of fruits.*

Fruit.	Coloration of acid solution. ^a		Coloration of ammoniacal solution.		Addition of a drop of H ₂ SO ₄ to dyed fabric.
	Juice.	Amyl-alcohol extract.	Juice.	Amyl-alcohol extract.	
Early cherries.....	Red	Yellow	Green	Uncolored	Yellow.
Ripe cherries.....	Red	Uncolored..	Green	Uncolored	Yellow.
Early strawberries ..	Red	Rose	Green	Uncolored	Rose.
Ripe strawberries ..	Red	Red	Green	Uncolored	Rose (tints silk a rose red.)
Raspberries.....	Red	Red	Green	Uncolored	
Red currants	Red	Uncolored..	Green	Uncolored	
White currants	White	Uncolored..	Brown.....	Uncolored	
Black currants	Dark red ..	Red	Deep green ..	Uncolored	Tints silk rose.
Peaches	Yellow	Uncolored..	Brown.....	Yellow red ...	Uncolored.
Pears.....	Yellow ..	Uncolored..	Brown.....	Yellow red ...	
Quinces	Yellow ...	Uncolored..	Brown.....	Yellow red ...	
Apples.....	Yellow ...	Uncolored..	Brown.....	Yellow red ...	
Apricots.....	Yellow ...	Uncolored..	Brown.....	Yellow red ...	
Green gage plums.....	Yellow ...	Uncolored..	Brown.....	Yellow red ...	

^a Acidity of the juice.TABLE 2.—*Coloring matter of fruits.*

Fruit.	Color with NH ₄ OH.	Ether extract from acid solution.	Amyl-alcohol extract from acid solution.	Dyeing tests on the juice.
Strawberry.....	Purple.....	None	Deep red	Color washed out.
Red raspberry.....	Purple.....	None	Deep red	All color does not wash out, but does not dye in the second acid bath.
Blackberry.....	Blue-purple ..	None	Very deep red.	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Cherry	Purple.....	None	Red	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Blackberry.....	Blue-purple ..	None	Red	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Wild dewberry	Blue-purple ..	None	Red	Dyes purplish red in acid solution, but does not dye in the second acid bath.
Currant.....	Blue-purple ..	None	Red	Dyes purplish red in acid solution, but does not dye in the second acid bath.

It will be seen from these two tables that amyl alcohol, as a rule, extracts fruit coloring matter from acid solution, while ether does not. Neither amyl alcohol nor ether extracted any color from alkaline solution of the fruit juices.

DETECTION OF ACID MAGENTA.^a

Add to 100 cc of the solution to be tested 2 cc of potassium hydroxid (5 to 100); if this does not neutralize the acid, add enough to do it. Then add 4 cc of mercuric acetate (10 to 100), agitate, and filter. The filtrate should be colorless and slightly alkaline. Acidify with a slight excess of dilute sulphuric acid, and if the solution remains uncolored there is no acid magenta present. If it becomes a light violet red and there has been no other dye shown by the amyl-alcohol extracts, the presence of acid magenta is shown.

Acid magenta dyes wool a magenta red from acid solution.

Wool dyed with it is turned yellow by strong hydrochloric acid, decolorized by ammonium hydroxid, and regains its color when washed with water.

DETECTION OF COCHINEAL.

Cochineal is used to a certain extent as a coloring matter in foods, and a very satisfactory test for it is that given in Girard and Dupré.^b Dissolve the food product in water, filtering if necessary. Acidulate with hydrochloric acid and extract with amyl alcohol, which becomes colored more or less yellow or orange, depending on the quantity of cochineal present. Separate the amyl alcohol and wash with water until neutral. Then separate into two portions; to the first add drop by drop a very dilute solution of uranium acetate, shaking thoroughly after each addition. In the presence of cochineal a characteristic emerald-green color is produced.

To the second portion add a drop or so of ammonia, and in presence of cochineal a violet coloration results. This, however, is not so sensitive to very small amounts as the first test, and many fruit colors give tests hardly to be distinguished.

Cochineal carmin is liable to contain tin, as it is often a tin lake, although alum is also used. It is also liable to adulteration with lead compounds.

DETECTION OF CARAMEL.

Amthor test.^c—Place 10 cc of the solution to be tested into a high, narrow glass with perpendicular sides, as, for example, a small bottle; add from 30 to 50 cc of paraldehyde, depending on the intensity of the coloring, and enough absolute alcohol to make the solutions mix. In the presence of caramel a brownish-yellow to dark-brown precipitate will collect in the bottom of the glass. Decant the liquor, wash once with absolute alcohol, dissolve in a small amount of hot water, and filter. The color of this will give some idea as to the amount of caramel present.

^a Girard and Dupré, *Analyse des Matières Alimentaires*, etc., p. 169; Winton, Conn. Expt. Sta. Rept. 1899, Pt. II, 132.

^b *Analyse des Matières Alimentaires*, etc., p. 580.

^c *Ztschr. anal. Chem.* 1885, **24**, 30; *Borgmann Anal. des Weines.*, p. 98.

It is not allowable to concentrate a solution by evaporation on a steam bath, as caramel may be formed; if it is necessary to concentrate, it must be done over sulphuric acid or at diminished pressure.

In order to further identify the color it is poured into a freshly prepared solution of phenylhydrazin (2 parts phenylhydrazin-hydrochlorid, 3 parts sodium acetate, and 20 parts of water). The presence of a considerable quantity of caramel gives a dark-brown precipitate in the cold, hastened by heating a little.

In the case of a very small amount it takes some hours for it to collect.

DETECTION OF GELATIN.

The presence of gelatin in jellies and jams is shown by a higher content of nitrogen. Precipitate a concentrated solution of jelly or jam with 10 volumes of absolute alcohol and determine nitrogen in dried precipitate by the Gunning method.^a

E. Beckmann^b recommends the following method for the determination of gelatin in jellies:

The jelly is treated with 95 per cent alcohol, the precipitate washed with alcohol to remove all sugar, and the alcohol finally removed by heating. The residue is taken up with water and the extract is neutralized with calcium carbonate and then treated with formalin. Upon evaporation to dryness the gelatin is rendered insoluble. With pure fruit jellies Beckmann found from 1 to 2 per cent of the precipitate insoluble, while with jellies to which gelatin had been added 70 to 86 per cent of the precipitate was found to be insoluble.

DETECTION OF AGAR AGAR.^c

Boil the jelly with 5 per cent sulphuric acid, add a crystal of potassium permanganate, and allow to settle. If agar is present the sediment will be rich in diatomes, which can be detected by use of microscope.

Bömer gives the following analyses of two marmalades, one of which contained agar and the other gelatin, which show how the addition of these substances affects the different constants:

TABLE 3.—*Marmalade containing agar and gelatin.*

	Solids.	Insoluble solids.	Ash.	Protein.	Acid as malic.	Invert sugar.	Cane sugar.	Pectins.	Polarization.
With agar	67.78	1.39	0.84	0.85	1.26	39.32	19.60	2.93	+3.64
With gelatin	70.96	1.31	.74	1.94	1.23	43.92	17.86	2.52	+1.27

^a A. Bömer, Chem. Ztg., 1895, **19**, 552.

^b Forschungsberichte über Lebensmittel, 1896, **3**, 327; Chem. der Nahr. und Genussmittel, 1896, **11**, 378.

^c G. Marpmann, Ztschr. angew. Mikrosk., 1896, **11**, 257.

The pectin precipitate had the following content of nitrogenous material:

	Pure marmalades.					With agar.	With gelatin.
	1.	2.	3.	4.	5.		
In per cent of substance.....	0.313	0.22	0.22	0.313	0.363	0.22	1.125
In per cent of pectins	28.2	17.6	20.7	13.3	16.5	7.6	44.6

DETECTION OF STARCH.

In the detection of starch it is first necessary to destroy the color of the fruit or fruit product. This is accomplished by bringing the solution of jelly or jam nearly to the boiling point, and after the addition of several cubic centimeters of dilute (1 to 3) sulphuric acid, adding potassium permanganate with constant stirring until the color disappears. By this treatment the starch remains unaffected, and the solution may be tested with iodine. If starch is found to be present it is well to examine the product microscopically in the case of jams and marmalades. If the starch is normally present in the fruit this point is easily decided by such an examination, as the starch grains may be readily detected within the cell walls after the treatment with iodine.

DETERMINATION OF HEAVY METALS.

Treat 100 grams of the preserve directly in a large porcelain evaporating dish with sufficient concentrated sulphuric acid to thoroughly carbonize the mass. If much water is present evaporate the material to a sirupy consistency before treating with the acid. From 10 to 15 cc of strong acid has been found sufficient to thoroughly carbonize the amount specified. Gently heat over a Bunsen burner until all danger of foaming is past, which will require not more than three minutes; then transfer the dish to a muffle furnace and keep it at a low red heat until all organic matter is destroyed. It is occasionally found necessary to add a few drops of nitric acid to completely destroy organic matter. When the material is completely ashed allow the dish to cool; add 25 cc of hydrochloric acid (1 to 8) and evaporate on a water bath to dryness; take up with water and acidify with two or three drops of hydrochloric acid. Transfer to a beaker without filtering and treat with hydrogen sulphide. After heating upon a water bath for a few minutes the precipitate and the insoluble residue are collected upon a filter. The precipitate and residue may contain sulphide of tin, lead, and copper, and oxide of tin; the filtrate will contain any zinc that is present. Fuse the sulphide precipitate and insoluble ash residue with about 3 grams of sodium hydroxide in a silver crucible for a half hour to render soluble any insoluble tin compounds. Dissolve the mass with hot water and slightly acidify with hydrochloric acid.

Again treat with hydrogen sulphid without filtering. By this treatment all the tin is thrown down as sulphid with the sulphids of copper and lead. Collect the precipitate upon a filter and wash thoroughly with hot water. The filtrate may be rejected. To separate the tin sulphid from those of copper and lead, wash several times upon the filter with separate portions of 10 cc of strong, boiling ammonium sulphid. Usually 50 cc of the ammonium sulphid will be found sufficient to completely dissolve all tin sulphid; but portions of the filtrate should be tested to make sure of this point. The filtrate is then made acid with hydrochloric acid to precipitate the tin sulphid, which, after standing for a few moments, is collected upon an ashless filter, ignited, and weighed as stannic oxid.

Treat the residue insoluble in ammonium sulphid with nitric acid, filter, wash, nearly neutralize with ammonia the excess of mineral acid, and add ammonium acetate, as there is usually a small amount of iron present. If any iron salt precipitates, filter, wash, and divide the filtrate for the determinations of copper and lead. In the absence of lead, copper may be determined electrolytically, or it may be titrated with potassium cyanid. Unless added as a coloring agent, copper will seldom be present in sufficient quantity to warrant its determination.

Precipitate lead with potassium chromate in an acetic acid solution, and weigh upon a tared filter as lead chromate.

Evaporate the filtrates from the hydrogen sulphid precipitate to about 60 cc; add bromin water to oxidize the iron salts and any remaining hydrogen sulphid. Boil off the excess of bromin and, unless the solution is distinctly yellow, add a few drops of concentrated solution of ferric chlorid to make it so. Nearly neutralize the mineral acid with ammonia, and add ammonium acetate to precipitate iron phosphate and excess of iron. Filter and thoroughly wash the precipitate. To the filtrate, made distinctly acid with acetic acid and boiled, add hydrogen sulphid to precipitate zinc. Unless the zinc sulphid comes down white it should be dissolved, again treated with ammonium acetate to remove traces of iron, and reprecipitated as sulphid. Finally collect the zinc sulphid upon an ashless filter, ignite, and weigh as zinc oxid.

ADDED SUBSTANCES.

GLUCOSE.

The substitution of glucose for the more expensive sugars is extensively practiced in the manufacture of various food materials, and in no class of foods is there better opportunity offered for this substitution than in fruit products. Of 214 samples of commercial fruit products examined, 110 samples contained glucose. The amount used varies

widely. Many samples of the better classes of goods contained not more than from 10 to 15 per cent, while some of the cheapest jellies were made up almost entirely of glucose, and many of the jams and marmalades had only small proportions of fruit and apparently no cane sugar. While glucose may be considered as an article of food, and as such has no deleterious properties, its use in substitution for a more expensive sugar must be considered a fraud unless its presence is indicated in some way to the purchaser. If low-priced goods are purchased it is not expected that they will be made up of first-class materials, and the use of glucose is the most efficient means of producing a cheap article. At the same time all goods should be of the quality represented on the label. Many of the high-priced fruit products bought upon the market were found to contain glucose. While the amount of glucose used in the better grade of goods was usually small, there was nothing upon the label to indicate that any glucose had been used in their preparation.

Considering the importance of glucose in the interpretation of the results given in this bulletin, a brief account of methods of manufacture practiced in the United States is given. According to Saare^a the raw product is always cornstarch. In the process of manufacture of sirups hydrochloric acid is used almost entirely as an inverting agent, but with sugars sulphuric acid may be used instead. The starch is mixed to a milk of about 22° to 23° Baumé and 0.75 part of concentrated hydrochloric acid per 100 parts of dry starch added. The mixture is then heated under from 2 to 2½ atmospheres pressure for a time sufficient to obtain the product desired. After inversion the material is drawn off into mixing tanks and the acid is nearly neutralized with sodium carbonate or a mixture of sodium and calcium carbonates. The salts obtained by neutralizing the above amount of acid would be about 0.25 to 0.30 per cent in the finished product. This thin sirup is filtered through presses and through charcoal, concentrated to about 30° Baumé, again filtered through charcoal, and finally concentrated to the desired consistency in vacuum pans. During the final concentration small amounts of acid sodium or calcium sulphite or sulphurous acid may be added to produce a light-colored product. The methods of procedure vary with the product to be obtained. Confectioner's glucose must give a decided starch reaction, and the percentage of reducing sugar must not be more than 48 per cent of the total solids indicated by the specific gravity. With brewer's glucose a high percentage of maltose is desirable; hence the dextrose will be low and the dextrin also high. Mixing glucose must give no starch reaction and the percentage of reducing sugars must be between 50 and 53 per cent of the

^aOscar Saare, *Industrie der stärke, und der Stärke-fabrication in der Vereinigen Staaten vom Amerika und ihr Einfluss auf den englischen Markt.*

solids indicated by specific gravity. In the manufacture of glucose sugars double the amount of acid used with the sirups is employed, and the inversion is continued until the dextrin reaction upon addition of alcohol is no longer shown. The sirups all have a high content of dextrin, and the reducing sugars present are variable mixtures of dextrose and maltose, the latter usually being in excess. The sugars have a low content of dextrin and maltose and a correspondingly high content of dextrose. The analysis, in this laboratory, of five representative samples of glucose sirups and sugars gave the following results:

TABLE 4.—*Composition of commercial glucose.*

Laboratory number.	Description.	Water.	Ash.	Reducing sugars as dextrose.	Polarizations.			Dextrose.	Maltose.	Dextrin.
					Direct.	Invert.	After fermentation 10 per cent solution.			
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Degrees.</i>	<i>Degrees.</i>	<i>Degrees.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
22009	Neutral glucose sirup.....	18.00	0.259	34.72	172.4	171.4	35.40	14.00	33.40	30.99
22010	XXX glucose.....	18.34	.459	36.40	167.6	167.6	30.30	14.53	38.24	26.53
22011	70 per cent sugar..	18.56	.837	69.82	66.00	66.0	3.30	68.71	1.79	2.89
22012	80 per cent sugar..	10.34	.796	79.84	73.8	73.8	4.20	79.28	.90	3.68
22013	Anhydrous glucose	6.18	.351	87.68	74.00	74.00	1.00	86.43	2.02	.88

In the above analyses the nonfermentable material is calculated as dextrin. This is not strictly correct, especially with the sirups, where small amounts of gallasin are also present. The two sirups are undoubtedly representative of the glucose sirups used with fruit products, although sirups having a much higher dextrin content may be used.

The composition of the ash of the above samples was as follows:

TABLE 5.—*Composition of the ash of glucose.*

Laboratory number.	Description.	Ash.	Chlorids.		Sulphates.		Lime.		Carbonates (soluble).	
			As per cent Cl.	As per cent NaCl.	As per cent SO ₃ .	As per cent K ₂ SO ₄ .	As per cent CaO.	As per cent CaSO ₄ .	As per cent Na ₂ CO ₃ .	As per cent K ₂ CO ₃ .
		<i>Per ct.</i>								
22009	Neutral glucose....	0.259	0.0368	0.0607	0.0269	0.0585	0.0207	0.0506	0.0702	0.0915
22010	XXX glucose.....	.459	.0764	.1258	.0416	.0905	.0081	.0196	.1426	.1859
22011	70 per cent sugar...	.837	.3962	.6528	.0654	.1422	.0669	.1624	.1008	.1314
22012	80 per cent sugar...	.796	.2024	.3334	.0846	.1840	.0781	.1895	.1165	.1519
22013	Anhydrous dextrose	.351	.0575	.0948	.0468	.1018	.0273	.0663	.1265	.1650

The following analyses, made by J. S. C. Wells,^a give the composition of the ash of a number of samples of American glucose:

TABLE 6.—*Composition of the ash of glucose.*

Mark.	Water.	Ash.	Sulphuric acid (SO ₃).	Chlorin.	Ferric oxid.	Lime.	Magnesia.	Alkalies.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Glucose.....	11.50	0.325	0.202	0.055	0.020	0.012	0.027	0.097
Mixing sirup.....	17.15	.370	.177	.060	.015	.008	.023	.140
Glucose.....	12.18	.520	.039	.255	.030	.066	.005	.157
Mixing sirup.....	24.25	.890	.220	.155	.015	.014	.031	.380
Glucose.....	14.50	.420	.161	.065	.010	.014	.025	.150
Brewer's grape sugar.....	12.98	.380	.055	.010	.055	.029	.038	.070
Confectioner's grape sugar.....	10.00	.140	.091	.010	.025	.025	.007	.017
Cakes from centrifugal.....	4.20	.220	.139	.020	.035	.035	.023	.016
Anhydrous grape sugar.....	.53	.025	.009	.000	.006	.004	.004	.000
Mixed grape and cane sugar...	1.75	.330	.029	.120	.025	.025	.004	.120
Grape sugar.....	13.25	.495	.230	.050	.100	.047	.004	.140
Do.....	11.55	.750	.395	.025	.030	.029	.004	.320
Glucose.....	23.10	.335	.094	.105	.010	.012	.009	.134
Grape sugar.....	16.50	.335	.159	.050	.055	.027	.004	.102
Maltose.....	22.26	1.060	.056	.125	.000	.021	.101	.337
Maize sirup.....	24.46	.815	.065	.120	.010	.008	.013	.374
Glucose.....	18.41	.335	.053	.025	.015	.080	.034	.012
Family sirup (mixed cane and grape).....	21.17	1.535	.094	.205	.040	.226	.088	.340

PRESERVATIVES.

Preservatives find an extensive use with those classes of fruit products put up in glass and not hermetically sealed. With fruits put up in tin or in sealed glass jars the use of any preservative would be superfluous if the fruit and the receptacle were properly sterilized and the latter properly sealed at the time of canning. It would seem, too, that their use for the purpose of preserving jellies and jams subsequent to their preparation should not be necessary, as the high content of sugar present is sufficient to prevent any fermentation. This point is borne out in the home preparation of jellies, jams, and marmalades where no preserving agent is used, and yet the danger of the product spoiling is very slight. Furthermore, those products which were found to contain no preservatives showed keeping qualities in no way inferior to those that had been artificially preserved. While many manufacturers undoubtedly use preservatives to prevent spoiling subsequent to the preparation of the articles, others use them for the purpose of keeping the fruit until it is worked up into the finished product. The pulped fruit to be used for making jellies and jams is sometimes kept for several weeks after the addition of some sugar,

^a Report on glucose to the Commissioner of Internal Revenue by The National Academy of Sciences.

but not sufficient to preserve it, before being worked up into the finished product. When this procedure is followed the use of some form of preservative is necessary. Canned fruits, on the other hand, are put up as fast as they are supplied to the factory, and hence need no artificial preservation.

Salicylic acid and benzoic acid or the sodium salts of these two acids are the preservatives most commonly employed with fruit products, and were found to be present in more than half of the jellies and jams examined, while the percentage of canned fruits found to be artificially preserved was comparatively small.

STARCH.

The presence of starch in fruit products may be attributed to one of two sources: It may have been added as such to jellies, and possibly jams and marmalades, in order to give them the proper consistency when converted by heat and moisture into paste, or it may have been normally present in the fruit used as a basis for the product. The ordinary means of detection afford no basis for determining to which source the starch found in jellies is due. With jams and marmalades from which portions of the fruit are still obtainable a microscopic examination will reveal whether the starch is normally present, as a larger part of the starch remains within the cell walls after the fruit has passed through the various stages of preparation. With jellies, however, if the amount of starch added has been small and the product has been thoroughly heated subsequent to its addition, or if the fruit from which the jelly was made contained starch, the starch, in the finished product, will be present in a soluble form. Only when an excessive amount has been added or when the boiling has not been sufficient to bring all the starch into solution can the addition be detected with certainty. Many jellies, of course, are made up purely artificially or with the use of only small amounts of fruit. In such cases the polarization and the per cent of dextrin present will reveal the character of the goods, and any appreciable amount of starch in such products may be considered as having been added.

Considerable amounts of starch are normally present in the apple, and probably some similar fruits, but the content gradually diminishes during ripening, and finally disappears in the thoroughly matured fruits. With the small fruits, on the other hand, starch is seldom found to be present in more than traces, even when these fruits are too green for use. As a result, pure apple jellies may contain considerable amounts of starch, while the pure jellies from the small fruits show no starch reaction. Not all pure apple jellies, however, contain starch, as the product may have been made subsequent to the complete

conversion of the starch. The following table of analyses of the Baldwin apple, by C. A. Browne,^a shows the changes that take place during the period of ripening:

TABLE 7.—*Composition of apples at various stages of maturity.*

Date.	Condition.	Solids.	Invert sugar.	Cane sugar.	Starch.	Acidity as per cent malic acid.	Ash.
1899.		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>
Aug. 7	Very green	18.47	6.40	1.63	4.14	1.14	0.27
Sept. 13	Green	20.19	6.46	4.05	3.67		
Nov. 15	Ripe	19.64	7.70	6.81	.17	.65	.27
Dec. 15	Overripe	19.70	8.81	5.26	None.	.48	.28

Of particular importance is the rapid disappearance of starch as the period of ripening progresses. Many of the pure apple jellies examined showed no starch reaction whatever, showing that the starch had already been converted. Hence the absence of starch in any jelly can not be taken as evidence that apple was not used as a basis.

Owing to its excellent jellying property the apple is very extensively used as the basis with many fruits that alone will not produce a jelly of proper consistency. This addition is very difficult of detection when a sufficient amount of the particular fruit is used to give the proper flavor. Where the apple has been used with the small fruits the presence of a small amount of starch will be an indication although not a proof of its presence, since, as above stated, added starch can not be differentiated. The use of apple with the large fruits in making jelly is undoubtedly rather limited, but its use is frequent in connection with the small fruits.

Of the jellies examined a large number were found to contain starch. Many of these give only slight reactions, indicating that the starch was normally present in the fruit. In only a few cases was it apparent that starch had been added in making up the product, and these were the cheapest grade of goods found on the market.

COLORING MATTER.

The use of foreign coloring material in fruit products is very widespread for two reasons. One is that the color of the fruit is not very stable, and the processes of preserving are liable to dim or destroy it, and, furthermore, the color will not last in goods that are constantly exposed to the action of the light, as is the case with those placed on the store shelves. The other reason is that it enables the manufacturer to use fruit of deficient color and thus to conceal inferiority. The

^a Pennsylvania Dept. of Agr. Bul. 58, p. 15.

preservation of this color is important, as the appearance of jellies and jams undoubtedly influences their real value, especially in the sick room, where they are used to a great extent, but the possibilities of deception as to the quality and purity which the addition of coloring matter affords entirely overbalance any argument in its favor. By the judicious use of coal-tar colors apple jelly flavored with currants can be given the appearance of the pure article, or a cheap fruit or a vegetable pulp can be mixed into a jam; a jelly made of glucose and starch may be served to consumers who demand pure goods. In most European countries certain stated colors are allowed in food products which have no natural color but are ordinarily colored commercially, such as candies, confections, liqueurs, and similar products, but great care has been taken in the selection of these colors to exclude any that may be injurious, either from being toxic in themselves or liable to contain injurious or poisonous impurities. Lists of the colors allowed by different countries have been published,^a and from these it would be possible to select harmless colors. The use in fruit products of colors of a vegetable origin is unquestionably nearly obsolete, as coal-tar colors are both cheaper and more durable. The latter are always liable to contain metallic impurities, such as zinc, copper, tin, lead, and arsenic retained during the process of manufacture, and which, when introduced into the food, even in the small quantities that are used, are, to say the least, a source of danger. Others of the coal-tar colors contain metallic atoms in their molecules—for example, malachite green, which is a double chlorid of zinc combined with the organic group. Even some of the vegetable colors are lakes of tin or some other metal. These facts all go to show that manufacturers should use great care in the selection of only the purest colors for use in food products.

GELATINIZING AGENTS.

Many fruits do not readily form a good jelly, especially when a little overripe, and it is necessary that some material be added in order to give the product the proper consistency. There are several articles that may be used to effect this result. Gelatin, agar, or some fruit which has a high gelatinizing power may be added. It has been claimed^b that gelatin is used, but in the large number of samples examined there was no indication that such was the fact. Gelatin is a very highly nitrogenized product, and even very small amounts would raise the nitrogen content far above the normal. Agar is another substance that might be used, and to much better advantage than gelatin, because of its very high gelatinizing power (a 1 per cent solution forms a stiff jelly) and because it is very similar in composition to

^aU. S. Dept. of Agr., Bureau of Chemistry Bul. 61, p. 9.

^bRept. Dairy and Food Commission, Minnesota, 1900, p. 102.

the pectin bodies of the fruits and would be much more difficult to detect if it were not for the fact that being obtained from seaweed it contains large numbers of diatoms, which can be readily detected by the use of the microscope. But it must be said that careful examination of the jellies described in this bulletin did not show the presence of this material. There can be no doubt that apple juice in some form is by far the most common substance used to give the desired consistency.

ARTIFICIAL SWEETENING MATERIALS.

Artificial sweeteners are used to some extent in fruit products, especially where glucose is used, in order to obtain the required degree of sweetness. These may get into the product in two ways. The manufacturer may add them, or he may use glucose which contains a sweetener.

There is said to be a mixture of crystallized dextrose, cane sugar, and saccharin on the market, which ordinarily would pass as cane sugar.

The sweetener generally used is saccharin (benzolsulphimid, or its sodium salt), and it has been claimed that sucrol (phenetolcarbamid) is also used, but it has never been noted in this country.

Saccharin is a coal-tar product of great sweetening power, claimed by the producers to be five hundred times sweeter than sugar, but it is not a sugar and has no value as a food. It is also a powerful antiseptic. It is sold in this country under various names, such as garantose, sugarine, suchrin, and suchrine. Sugarose is another product on the market which is a mixture of about 20 per cent of saccharin and 80 per cent of cane sugar.

Another curious product is a sirup made of glycerin in which a large amount of saccharin is dissolved.

In many of the European countries the use of saccharin is prohibited in food stuffs of all kinds.^a

The physiological action of both saccharin and sucrol have been studied somewhat extensively, and the literature on the subject collected.^b

FOREIGN FRUITS.

Aside from the use of apples as a basis for jellies, and occasionally jams, the substitution of one fruit for a more expensive one is but little practiced in this country. In many of the cheaper jams inferior fruits were used; in others only small amounts of any fruit were used; but in only one case substitution other than that above stated was detected. One sample of currant jam contained a large number of

^a U. S. Dept. of Agr., Bureau of Chemistry Bul. No. 61, p. 8.

^b Ver. Nahr. Genuss, Deutsche Reich, Heft II, pp. 137-142.

raspberry seeds, indicating that raspberry pulp had been used as the basis of the jam. It is said that in Europe the fig serves as the basis of a large number of jams, but its use can easily be detected by examination of the seeds. It is probably true that the exhausted pulp left from the manufacture of jellies is sometimes used as a basis for the poorer grade of jams.

HEAVY METALS.

Tin is almost universally present in goods put up in tin cans, owing to the ease with which the metallic surfaces are attacked. Canned fruits as a rule dissolve a larger amount of tin than other classes of foods, not alone because of their high acidity, but because the sirup in which the fruits are put up allows better contact with the walls of the can and better diffusion of the acids than is possible in many other classes of foods, such as meats, fish, etc.

The frequent occurrence of zinc in canned foods makes the determination of this metal of particular importance. The zinc present in the product is undoubtedly due in most cases to the zinc chlorid used as a flux in soldering. Since this salt may readily be removed by washing after the cans are made, care should be taken by the manufacturer that necessary precautions are used to remove this objectionable material. This point is well illustrated by the work of Hilgard and Colby,^a in which the presence of an excessive amount of zinc chlorid in canned asparagus was traced to the use of soldering fluid. By employing care in the manufacture of the cans and by careful washing before using, the manufacturers were able to reduce the amount of zinc to an inappreciable quantity.

The following table gives the results of the determination of heavy metals in 25 samples of fruit products:

TABLE 8.—*Percentage of heavy metals in fruit products.*

Serial number.	Kind of fruit.	Acidity of fruit as per cent H_2SO_4 .	Metals, milligrams per kilogram.	
			Tin.	Zinc.
20232	Apple jelly	0.592	None.	None.
19900	Apricots	.882	264	None.
20027	Cherries	.380	269	None.
20248	Cherries	.823	168	Trace.
19895	Cherries	.702	458	Trace.
19898	Cherries (black)	.401	216	None.
19881	Currants	.457	224	None.
19958	Currant and raspberry	.477	122	None.
19891	Cranberries	.455	429	28
20231	Grapes	.303	400	16
20233	Peaches		96	45
19893	Peaches	.485	176	11
20134	Pears	.205	133	104

^a California Experiment Station Report, 1897-98, p. 159.

TABLE 8.—*Percentage of heavy metals in fruit products—Continued.*

Serial number.	Kind of fruit.	Acidity of fruit as per cent H_2SO_4 .	Metals, milligrams per kilogram.	
			Tin.	Zinc.
20168	Pears		1,259	32
20140	Pears	0.137	214	Trace.
20144	Pears	.161	106	26
20028	Plums	.289	232	Trace.
19894	Plums	.769	232	108
20238	Plums	.602	213	None.
20139	Plums	.710	342	None.
20234	Pineapples	.509	563	28
19896	Pineapples	.563	252	None.
20228	Pineapples		41	None.
19879	Raspberries	.328	145	None.
19959	Raspberries	.412	139	None.

Tin was found in all cases except the one sample of apple jelly, and here the consistency of the product was not such as to favor an attack of the metal. Thirteen samples, or more than 50 per cent, contained zinc. While in most cases the amount is small and could not be considered harmful, Nos. 19394 and 20134 have an excessive amount of this harmful metal. Neither lead nor copper was found in any of the samples. The former is not commonly found in appreciable amounts in tinned goods, and the latter is seldom present in more than traces except where it has been added as a coloring agent.

There is no direct relation between the acidity of the product and the content of tin. Such a relation is not to be expected, since the time of heating for the purpose of sterilizing varies materially with different products, and the quality of tin used is not always the same. Hilgard and Colby showed in their work that the acid-finish tin plate was much more susceptible to attack than the oil-finish plate.

Sample No. 20168, which contained 1,259 mg per kilogram of tin or 0.126 per cent, was put up in an ordinary tin pail with cover. In this case the poorer grade of tin plate was undoubtedly used, which accounts for the excessive amount dissolved.

Sample No. 20228, which contained 40.9 mg. of tin per kilogram, was put up in glass and only the tin cover came in contact with the contents of the receptacle.

ANALYTICAL DATA.

FRESH FRUITS AND FRUIT JUICES.

TABLE 9.—*Composition of fresh fruits.*^a

Fruit and analyst.	Number of samples.	Total solids.	Ash.	Acidity expressed as H ₂ SO ₄ .	Protein (N × 6.25).	Reducing sugar.	Cane sugar.	Crude fiber.
Apples:								
G. E. Colby— ^b		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Average	13	{ 13.77	0.240	0.376	0.590	7.04	4.59	
Maximum		{ 16.47	.320	.670	.806		7.79	
Minimum		{ 9.37	.170	.190	.356		1.80	
C. A. Browne— ^c								
Average	27	{ 16.43	.27	.486		7.92	3.99	
Maximum		{ 23.36	.34	.811		11.75	6.81	
Minimum		{ 13.46	.17	.073		5.34	1.74	
Edgar Richards— ^d								
Average	23	{ 13.65	.288	.452	.694	8.73	1.53	0.96
Maximum		{ 16.55	.404	.863	1.094	10.80	2.81	1.29
Minimum		{ 10.60	.228	.139	.421	6.89	.15	.70
F. König ^e	17	16.42	.310	.614	.39	7.73		1.98
H. Kremla— ^f								
Average	5	{ 15.07	.290	.234		10.12	.55	
Maximum		{ 16.03	.360	.329		10.69	1.11	
Minimum		{ 14.04	.240	.190		9.77	None.	
Apricots:								
G. E. Colby— ^b								
Average	11	{ 14.34	.491	.833	1.250	11.10		
Maximum		{ 15.75	.558	1.090	1.040	12.50		
Minimum		{ 12.75	.450	.560	.897	9.66		
F. König ^e	6	18.78	.820	.848	.49	4.69		≠ 5.27
Blackberries: F. König ^e	1	13.59	.48	.870	.51	4.44		5.21
Cherries:								
G. E. Colby— ^b								
Average	6	{ 20.13	.443	.432	1.425	11.10		
Maximum		{ 38.84	.521	.605	1.727	12.75		
Minimum		{ 11.46	.403	.328	1.100	8.98		
F. König ^e	9	19.74	.73	.665	.620	10.24		≠ 6.07
Moleschott ^h	1	22.30	.65	.746	.810	11.72		.62
H. Kremla ^f	1	21.95	.38	.965		13.32	None.	
Currants: F. König ^e	7	15.23	.72	1.57	.51	6.38		4.57
Figs:								
G. E. Colby— ^b								
Average	41	{ 20.13	.578	.119	1.344	15.51		
Maximum		{ 38.84	1.160	.291	2.587	20.99		
Minimum		{ 11.46	.364	.073	.725	8.00		
Oranges:								
G. E. Colby— ^b								
Average	12	{ 13.87	.430	.672	.486	10.66	5.25	
Maximum		{ 14.71	.460	.714	.512	12.33	6.75	
Minimum		{ 13.20	.370	.518	.460	9.92	4.80	

^a From other laboratories.^b Calif. Expt. Sta. Rept., 1897-98, p. 143.^c Penn. Dept. of Agr. Bul. 58, p. 20.^d U. S. Dept. Agr. Rept., 1886, p. 354.^e Menschlichen Nahrungs. u. Genussmittel, p. 397.^f Ztschr. Nahr., Hyg. Waar., 1892, 6, 483.^g Includes pits.^h Des aliments hygiene et régimes alimentaires.

TABLE 9.—*Composition of fresh fruits—Continued.*

Fruit and analyst.	Number of samples.	Total solids.	Ash.	Acidity expressed as H ₂ SO ₄ .	Protein (N×6.25).	Reducing sugars.	Cane sugar.	Crude fiber.
Pears:		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per ct.</i>
F. König ^a	9	16.97	0.31	0.146	0.360	8.26		4.30
Moleschott ^b		16.77	.35	.022	.230	8.78		2.77
H. Kremla ^c	2	20.12	.45	.140		9.00	4.36	
Plums:								
F. König ^a	3	15.14	.61	1.096	.40	3.56		4.34
G. E. Colby ^d	3	21.60	.524	.996		13.25		
H. Kremla ^c	1			1.257		2.57	3.56	
Raspberries: F. König ^a	4	13.79	.490	1.010	.53	3.95		5.90
Strawberries:								
F. König ^a	33	12.34	.810	.68	1.070	6.28		2.32
H. Kremla ^c	1	13.67		.965		6.36	None.	
W. E. Stone— ^e								
Average.....	20	9.48	.606	1.001	.974	4.78	.58	1.518
Maximum.....		12.28	.818	1.389	1.232	6.71	1.17	2.266
Minimum.....		7.57	.372	.768	.723	3.91	.02	1.036

^a Menschlichen Nahrungs. u. Genussmittel, p. 397.^d Calif. Expt. Sta. Rept., 1897-98, p. 143.^b Des aliments hygiene et régimes alimentaires.^e Tenn. Expt. Sta. Bul., Vol. II, No. 4, pt. 2.^c Ztschr. Nahr., Hyg. Waar., 1892, 6, 483.TABLE 10.—*Composition of fresh fruit juices.^a*

Fruit and analyst.	Number of samples.	Specific gravity.	Total solids.	Ash.	Acidity expressed as H ₂ SO ₄ .	Protein (N×6.25).	Reducing sugar.	Cane sugar.
Apples:			<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per ct.</i>
C. A. Browne— ^b								
Average.....	11	1.0553	13.18	0.279	0.490		7.86	3.56
Maximum.....		1.0736	16.82	.370	.906		10.52	7.05
Minimum.....		1.0488	11.36	.240	.073		5.47	1.63
Truchon and M. Claude ^b		1.0680		.340	.450		9.62	.61
Blackberries: A. L. Winton ^c	1		8.94		.854		8.70	None.
Cherries (red):								
Trucho and M. Claude ^b	1	1.055		.367	.510		9.14	None.
H. Kremla— ^d								
Average.....	8	1.0862	17.84		.351		12.25	
Maximum.....		1.1023	19.65		.497		13.40	
Minimum.....		1.0639	15.61		.212		9.50	
W. Keim ^e	2	1.0517	17.57	.559	.409		12.86	
Cherries (black): H. Kremla ^d	2	1.1019	24.12		.493		15.50	
Currants:								
Truchon and M. Claude ^b	1	1.0650	16.25	.670	.206		10.96	None.
W. Keim ^e	1	1.0425	14.46	.560	.172		5.24	
H. Kremla— ^d								
Average.....	9	1.0518	12.71	.518	1.805	0.440	6.91	
Maximum.....		1.0644	15.70	.758	2.330	.711	8.22	
Minimum.....		1.0400	9.90	.356	1.477	.248	4.61	
Grapes: G. E. Colby ^f			23.70	.300	.377		22.23	
Do.....	6		21.05	.210	.422		21.05	
Do.....	9		23.47	.370	.234		23.14	
Do.....	6		21.68	.230	.396		19.82	

^a From other laboratories.^d Ztschr. Nahr., Hyg. Waar., 1892, 6, 483.^b Journ. Pharm. Chem., 1901, vol. 13, p. 171-176.^e Ztschr. anal. chem., 1891, 30, 401.^c Conn. Expt. Sta. Report, 1899, pt. 2, p. 127.^f Viticultural Report, Cal. Expt. Sta., 1887-1893.

TABLE 10.—*Composition of fresh fruit juices—Continued.*

Fruit and analyst.	Number of samples.	Specific gravity.	Total solids.	Ash.	Acidity expressed as H ₂ SO ₄ .	Protein (N×6.25).	Reducing sugar.	Cane sugar.
			<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Huckleberries: A. L. Winton ^a .	1		11.40		0.350		16.70	0.60
Peaches:								
A. L. Winton ^a			12.70		.595		2.10	5.40
Truchon and M. Claude ^b		1.0540	13.61	0.446	.421		3.18	1.87
Pineapples: A. L. Winton ^a			13.90		.476		9.10	7.40
Raspberries:								
A. L. Winton ^a			9.41		.833		8.60	.80
Truchon and M. Claude ^b		1.050	12.63	.411	1.100		8.39	None.
Strawberries: Truchon and M. Claude ^b —								
Ripe		1.048	12.14	.546	.856		9.54	None.
Early		1.026	6.71	.580	.576		5.37	None.
Quinces: Truchon and M. Claude ^b	1.	1.0480	12.14	.400	.285		7.24	None.

^a Conn. Expt. Sta. Report, 1899, pt. 2, p. 127.^b Journ. Pharm. Chem., 1901, vol. 13, p. 171-176.

The composition of the pure fruits and fruit juices used as the basis of the fruit products is of great value in interpreting the results obtained in the analysis of these various materials. To meet this need Tables 9 and 10 have been compiled, the first giving the averages of analyses of whole fruits, the second of fruit juices. In addition, a large number of analyses of the fresh fruits and juices have been made by the writers, results of which are given in Tables 11 and 12. While the analyses compiled in Tables 9 and 10 are incomplete in many respects, they give a fair idea of the proximate composition of nearly all the fruits dealt with in the bulletin. It was not attempted to make this compilation complete. Only such analyses as seemed of particular value in aiding the interpretation of analytical data are included. It must be remembered that these analyses are of samples from extremely variable sources, and as a rule represent a large number of varieties, hence a wide variation in their composition is to be expected.

TABLE 11.—*Description of fresh fruits and juices.*

Serial number.	Description.	Variety.	From whom purchased.
	BLACKBERRY.		
22719	Whole fruit	Wild dewberries	White Bros., Center Market, Washington, D. C.
22743	do	Probably Erie	C. Engel & Son, Center Market, Washington, D. C.
22745	do	Early Harvest	F. R. Cooke, Center Market, Washington, D. C.
22718	Juice	Wild dewberries	White Bros., Center Market, Washington, D. C.
22742	do	Probably Erie	C. Engel & Son, Center Market, Washington, D. C.
22744	do	Early Harvest	F. R. Cooke, Center Market, Washington, D. C.

TABLE 11.—*Description of fresh fruits and juices*—Continued.

Serial number.	Description.	Variety.	From whom purchased.
	CHERRY.		
22747	Whole fruit	Morella Seeßling	J. M. Abbott, Center Market, Washington, D. C.
22746	Juice	do	Do.
	CURRENT.		
22717	Whole fruit		Rice Bros., Center Market, Washington, D. C.
22716	Juice		Do.
	RASPBERRY (BLACK).		
22695	Whole fruit	Probably Doolittle ...	Rice Bros., Center Market, Washington, D. C.
22697	do	Probably Gregg	F. R. Cooke, Center Market, Washington, D. C.
22694	Juice	Probably Doolittle ...	Rice Bros., Center Market, Washington, D. C.
22696	do	Probably Gregg	F. R. Cooke, Center Market, Washington, D. C.
	RASPBERRY (RED).		
22691	Whole fruit		Rice Bros., Center Market, Washington, D. C.
22693	do		Experiment Station, College Park, Md.
22690	Juice		Rice Bros., Center Market, Washington, D. C.
22692	do		Experiment Station, College Park, Md.
	STRAWBERRY		
22363	Whole fruit	Lovett	Experiment Station, College Park, Md.
22365	do	Tennessee Prolific	Do.
22367	do	Buback	Do.
22369	do	Clyde	Do.
22370	do	Wild strawberry	Office of Experiment Stations.
22362	Juice	Lovett	Experiment Station, College Park, Md.
22364	do	Tennessee	Do.
22366	do	Buback	Do.
22368	do	Clyde	Do.

TABLE 12.—Composition of fresh fruits and juices.

Serial num-ber.	Description.	Solids.		Protein (N×6.25).	Acidity expressed as H ₂ SO ₄ .	Sugars.		Polarizations.			Alcohol precip-itate.	Sugar-free soluble solids.	Ash.		
		Total.	Insol-uble.			Reduc-ing sugar.	Cane sugar.	Total.	Direct.	Invert.			Tem-perature.	Total.	Alka-linity as K ₂ CO ₃ .
		Per ct.	Per ct.	Per cent.	Per cent.	Per ct.	Per ct.	Per ct.	° V.	° V.	° C.	Per ct.	Per ct.	Per ct.	Per ct.
BLACKBERRY.															
22719	Whole fruit.....	13.65	6.24	0.863	0.548	4.69	None.	4.69	+0.6		25	0.736	0.448	0.429	0.001
22743	do.....	12.06	5.40	.788	.808	4.74	None.	4.74	+ .4		25	.700	.823	.634	Trace.
22745	do.....	12.37	4.72	1.106	.558	4.57	.49	5.06	± 0		25	.608	.484	.382	.003
	Average.....	12.69	5.45	.919	.638	4.67	.16	4.83	+ .3		25	.681	.585	.482	.001
	Maximum.....	13.65	6.24	1.106	.808	4.74	.49	5.06	+ .6			.736	.823	.634	.003
	Minimum.....	12.06	4.72	.788	.548	4.57	None.	4.69	± 0			.608	.448	.382	Trace.
22718	Juice from 22719.....	6.96		.069	.573				± 0		25		.423	.411	.032
22742	Juice from 22743.....	7.14		.319	.835								.346	.357	Trace.
22744	Juice from 22745.....	7.80		.350									.377	.354	.005
	Average.....	7.30		.246	.704								.382	.374	.030
	Maximum.....	7.80		.350	.835								.423	.411	.032
	Minimum.....	6.96		.069	.573								.346	.354	Trace.
CHERRY.															
22747	Whole fruit.....	12.64	2.04	.650	1.627	6.84	None.	6.84	+ .2		26	.668	.602	.481	Trace.
22746	Juice from 22747.....			.388	1.465								.553	.524	Trace.
CURRANT.															
22717	Whole fruit.....	12.97	6.90	1.369	1.546	3.44	None.	3.44	1.4		26	.804	.602	.437	.003
22716	Juice from 22717.....	6.71		.300	1.642				-1.2		26		.454	.390	.003
RASPBERRY, BLACK.															
22695	Whole fruit.....	18.67	8.59	1.125	.774	6.39	None.	6.39	-3.0	-3.8	28		.770	.538	Trace.
22697	do.....	21.97	11.23	1.299	.774	6.59	None.	6.59	-3.6	-3.5	28		.854	.803	Trace.
	Average.....	20.32	9.91	1.212	.774	6.49	None.	6.49	-3.2	-3.6			.812	.670	Trace.
22694	Juice from 22695.....	11.17		.194	.808	7.28	None.	7.28					.633	.596	Trace.
22696	Juice from 22697.....	12.04		.312	.779	7.85	None.	7.85					.741	.696	.002
	Average.....	11.60		.253	.794	7.56	None.	7.56					.687	.646	.001

TABLE 12.—Composition of fresh fruits and juices—Continued.

Serial num-ber.	Description.	Solids.		Protein (N × 6.25).	Acidity expressed as H ₂ SO ₄ .	Sugars.		Polarizations.				Sugar- free soluble solids.	Ash.			
		Total.	Insolu- ble.			Reduc- ing sugar.	Cane sugar.	Total.	Direct.	Invert.	Tem- pera- ture.		Total.	Alka- linity as K ₂ CO ₃ .	Sul- phates as K ₂ SO ₄ .	Chlo- rides as NaCl.
	RASPBERRY, RED.	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>°V.</i>	<i>°V.</i>	<i>°C.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
22691	Whole fruit.....	13.16	6.62	.931	1.112	3.52	0.76	4.28	-1.0	-1.1	28	2.26	0.551	0.429	0.040	Trace.
22693	do.....	13.41	7.12	1.019	1.097	3.53	.83	4.36	- .6	-1.8	28	.696	.527	.415	.043	Trace.
	Average.....	13.28	6.87	.975	1.104	3.52	.80	4.32	- .8	-1.4	28	.736	.539	.422	.042	Trace.
22690	Juice from 22691.....	6.92		.406	1.127	3.65			- .4			.655	.522	.422	.031	Trace.
22692	Juice from 22693.....	7.03		.388	1.112	3.64			- .8			.728	.512	.384	.047	Trace.
	Average.....	6.98		.397	1.120	3.64			- .6			.692	.517	.403	.039	Trace.
STRAWBERRY.																
22363	Whole fruit.....	8.18	2.80	.600	.918	3.94	.04	3.98	-1.3	-1.4	28	.494	.582	.387	.043	.007
22365	do.....	8.22	3.01	.613	.911	3.25	.26	3.51	-1.1	-1.2	28	.524	.538	.411	.035	.019
22367	do.....	7.55	2.88	.469	.808	3.22			-1.0	-1.2	28	.560	.515	.336	.027	.004
22369	do.....	6.85	2.57	.513	.737	2.72	.09	2.81	- .9	-1.0	28	.477	.704	.347	.044	.005
22370	do.....	12.88	5.67	.744	1.200	2.98	1.43	4.41	-1.0	-1.4	28		.758	.705	.032	Trace.
	Average.....	8.74	3.39	.588	.915	3.22	.46	3.68	-1.1	-1.2	28	.514	.619	.437	.036	.007
	Maximum.....	12.88	5.67	.744	1.200	3.94	1.43	4.41	-1.9	-1.0	28	.560	.758	.705	.044	.019
	Minimum.....	6.85	2.57	.469	.737	2.72	.09	2.81	-1.3	-1.4	28	.477	.515	.336	.027	.004
22362	Juice from 22363.....	7.04		.100	.887	3.94	.11	4.04	-1.2	-1.0	28	.689	.496	.385	.023	.008
22364	Juice from 22365.....	6.09		.113	.916	3.52	.21	3.73	-1.1	-1.2	28	.518	.500	.309	.033	.012
22366	Juice from 22367.....	6.02		.144	.789	3.15	.20	3.35	-1.0	-1.2	28	.480	.486	.359	.035	.018
22368	Juice from 22369.....	5.33		.106	.744	2.59	.40	2.99	- .7	- .8	28	.541	.488	.319	.029	
	Average.....	6.12		.116	.834	3.30	.23	3.70	-1.0	-1.0	28	.557	.492	.343	.030	.013
	Maximum.....	7.04		.144	.916	3.94	.40	4.04	- .7	- .8	28	.689	.500	.385	.035	.018
	Minimum.....	5.33		.100	.744	2.59	.11	2.99	-1.2	-1.2	28	.480	.486	.309	.023	.008

Table 12 includes the analyses made in this laboratory of both fruits and juices. In the preparation of these samples the edible portion of the fruit was finely ground by passing through a food chopper, and a portion of this was saved for the analysis of the whole fruit. The other portion was placed in a jelly bag and the juice removed by pressure. Unless the juice came through clear it was filtered or allowed to stand and then decanted.

The table is so arranged as to best permit of comparison between the analyses of the fruits and the juices. The following points are of interest regarding the analytical results:

Solids.—The percentage of insoluble solids varies widely with different fruits, the lowest being 2.04 per cent in case of the cherry, and the highest 11.23 per cent in one of the samples of black raspberries. The insoluble material consists principally of fiber, although it also contains a small amount of ash and considerable nitrogenous material. The percentage of soluble solids in the whole fruit is usually considerably lower than the percentage of total solids in the corresponding juice on account of the concentration brought about in the separation of the juice.

Ash.—The content of ash also varies widely with the various fruits, but is fairly constant in the different samples of the same fruit. Invariably the whole fruits have a content of ash appreciably higher than the corresponding juices, indicating that a portion of the ash remains in the insoluble material, probably in the form of calcium salts. The composition of the ash of pure fruits and fruit juices is in some respects characteristic. As will be seen from the table, it consists largely of carbonates (sodium, potassium, calcium, and magnesium carbonates), while sulphates and chlorids are seldom present in appreciable amounts. With the amount of material taken for the determination of ash there is seldom more than a trace of either sulphates or chlorids. The alkalinity of the ash of pure fruit products is also characteristic, and this feature may be taken as an indication of the purity of the goods. Calculated as potassium carbonate, the average alkalinity of the ash of the fruits is 77 per cent of the total ash and of the juices 84.3 per cent of the total ash.

Acidity.—No attempt was made to separate and determine quantitatively the various organic acids, although it is well known that the acidity of the greater number of fruits is due not to a single organic acid, but to mixtures of organic acids and acid salts. The total acidity, therefore, was determined and results expressed as sulphuric acid (H_2SO_4). The acidity is exceedingly variable with different fruits, and even with different varieties of the same fruit.

Protein.—The nitrogenous material of the fruit is largely in an insoluble form. In the one sample of cherries examined the protein in the whole fruit was not greatly in excess of that in the juice, while in

all the other fruits the relative amount was much larger, and in case of the strawberry the juice contained only one-fifth of the amount present in the whole fruit. This point is of value in the examination of jellies where the use of gelatin as a gelatinizing agent is suspected. The average content of protein in 14 samples of jellies (see Table 19) prepared in the laboratory was 0.215 per cent, and the extremes were 0.418 per cent and 0.069 per cent; and of 19 pure samples bought on the market the average content of protein was 0.205 per cent and the extremes 0.312 and 0.112 per cent. The use of gelatin would raise the protein content far above the normal.

Sugars.—The sugars normally present in the fruits are dextrose, levulose, and cane sugar. As is indicated by the polarizations made at 86°, dextrose and levulose are present in nearly equal proportions; frequently there is an appreciable excess of levulose, but seldom an excess of dextrose. Cane sugar is an extremely variable factor, and may range from none in many of the samples reported to several per cent. As a general rule, the large fruits, such as the apple, pear, peach, plum, and orange, have a much higher content of cane sugar than the small fruits, such as the strawberry, raspberry, blackberry, and currant. In apples Colby reported an average of 4.59 per cent, a maximum of 7.79 per cent, and a minimum of 1.80 per cent; Browne reported an average of 3.99 per cent, a maximum of 6.81 per cent, and a minimum of 1.74 per cent, while Richards gives 1.53 per cent as the average and limits of 2.81 per cent and 0.15 per cent. In oranges Colby gives an average of 5.25 per cent and limits of 6.75 per cent and 4.80 per cent, and results reported for oranges, plums, peaches, and pineapples are also high.

Stone found, in 20 samples of strawberries, the average percentage of cane sugar to be 0.58, the maximum 1.17, and the minimum 0.02. In this laboratory an average of 0.46 per cent was found in the same fruit; in blackberries, 0.16 per cent; cherries, none; currants, none; black raspberries, none, and red raspberries, 0.80 per cent. Results given in Tables 14 and 15 also verify the general statement.

Polarization.—Table 13 gives the polarizations of a number of pure fruits and fruit juices, together with the content of reducing and cane sugars. The cane sugar was calculated from the polarizations by the

Clerget formula, $S = \frac{(a-b) 100}{144 - \frac{t}{2}}$. Here all the large fruits except the

apple and the pear show a dextrorotatory direct reading, while all the small fruits show a levorotatory direct reading. The invert readings at room temperature were all negative with the single exception of the plum, which also has a positive reading at 86°, indicating an excess of dextrose over levulose. The polarizations on all the other samples indicated either a mixture of equal parts of dextrose and levulose, or an excess of levulose. There is no direct relation between

either the direct or the invert reading at room temperature and the percentage of reducing sugar, owing to the variable amounts of cane sugar, but the invert polarization is in a general way indicative of the amount of total sugars. The approximate percentage of levulose in excess of dextrose may be figured from the polarization at 86° by multiplying the degrees of levorotation by 0.42 in case the normal weight has been used for the polarizations.

TABLE 13.—*Polarization of fruits and fruit juices.*

Laboratory number.	Description.	Direct polarization.	Invert polarization.	Temperature.	Invert polarization at 86°.	Per cent of invert sugar.	Per cent of cane sugar.
	JUICES.			°C.			
20400	Apple	-3.0	-4.6	18	-2.9	4.00	1.18
20396	Blackberry	-1.5	-1.6	18	-1.1	4.34	
	do. ^a	-2.4	-2.4	30	-0.0	8.70	
20404	Crab apple	-1.0	-2.4	18	-1.1	2.56	1.03
20401	Grape	-1.2	-2.4	18	-0.6	5.10	.89
20397	Huckleberry	-3.2	-4.4	18	-0.9	11.21	.89
	do. ^a	-4.0	-4.8	30	-1.0	16.70	.60
20430	Orange	+1.8	-1.3	18	0.0	1.52	2.29
20427	Peach	+4.0	-2.2	18	-0.7		4.59
	do. ^a	+4.8	-2.2	28	0.0	2.10	5.40
20431	Pear	+4.8	-6.4	18	-4.0	5.87	1.18
20429	Pineapple	+8.4	-3.7	18	-1.1	2.74	8.96
20426	Pineapple husk	+4.1	-2.3	18	-.7		4.73
	Pineapple ^a	+4.7	-4.8	28	-.8	9.10	7.40
20399	Plum	+2.0	+1.3	18	+2.4	4.86	.51
20402	do.	+1.4	-2.4	18	-1.8	2.87	2.81
	Red raspberry ^a	-1.6	-2.8	28	0.0	8.60	.90
22362	Strawberry	-1.2	-1.0	28		3.94	.15
22364	do.	-1.1	-1.2	28		3.52	.21
22366	do.	-1.0	-1.2	28		3.15	.20
22368	do.	-.7	-.8	28		2.59	.40
	WHOLE FRUIT.						
20449	Apple	-2.2	-3.6	18	-1.8	4.13	1.03
20415	Blackberry	-1.1	-1.0	18	.0	5.67	.00
20418	Crab apple	-.7	-3.0	18	-.8	5.68	1.70
	Grape	-1.6	-2.0	18	.0	1.11	.29
20425	Orange	+2.0	-2.5	18	-.8	4.13	3.33
20444	Pineapple	+1.0	-3.2	18	0.0	8.12	3.11

^a A. L. Winton, Conn. Expt. Stat. Report, 1899, Pt. II, p. 127.

A number of the fresh fruits were fermented to determine whether any nonfermentable optically active bodies were present. It was found that in all cases the nonfermentable matter was inappreciable and that with a 10 per cent solution the polarization after fermentation was never more than three or four tenths of a degree either to the right or the left.

Alcohol precipitate.—The alcohol precipitate consists largely of pectin bodies, and the determination is valued only for the estimation

of these substances. Starch, if present, will be thrown down here, as well as acid potassium tartrate and the calcium salts of organic acids. The acid potassium tartrate may be estimated by titrating the alkalinity of the water-soluble ash of the alcohol precipitate after ignition. The appearance of the solution as the alcohol is added in making the alcohol precipitate is a valuable indication as to the presence or absence of glucose or starch. In pure fruit products there is no turbidity, but the pectin bodies are thrown down in a flocculent mass, while if glucose is present there is a white turbidity and the precipitate is gummy.

The test for starch was made upon all the fruits given in Table 12, and in no case was there sufficient present to give an appreciable iodine reaction. This point is of particular importance in its bearing upon the examination of jellies, where apple is so frequently used as the basis. If it is safe to assume that starch is never present in appreciable amounts in these fruits—and the work done in this laboratory seems to warrant the assumption—then the presence of appreciable amounts of starch in the fruit jelly must be due either to apple or to added starch.

Attention is called to the composition of No. 22370, which is a sample of wild strawberry. In total solids, soluble solids, ash, cane sugar, proteids, and acidity it runs much higher than the four samples of cultivated strawberries, and in these respects, with exception of proteids, is higher than the average of the 20 analyses reported by Stone. Reducing sugars alone are lower than in the cultivated varieties.

JAMS AND JELLIES PREPARED IN THE LABORATORY.

Preliminary to the examination of the commercial fruit products it was thought desirable to have the analyses of a number of jams and jellies of known origin. Accordingly several different kinds of each product were prepared and were analyzed together with the pulped fruit and the juices from which they were made. The results of this investigation are given in Tables 14, 15, 16, and 17. The fruits were prepared by heating with sufficient water to prevent scorching until thoroughly softened and then well pulped. The juices were obtained by draining the pulped fruit, as above prepared, in a jelly bag. Neither the fruit nor the juice, therefore, is normal in its content of water, but is rather the material used as the basis for the jams and the jellies. The jams were prepared by adding approximately one part of sugar to two parts of crushed fruit, heating to boiling, and maintaining at this temperature for about twenty minutes. In the preparation of the jellies approximately equal parts of cane sugar and the strained juice were used and were heated to the point of boiling, which required about twenty minutes. In all cases the cane sugar, the fruit or juice, and the finished product were weighed, and from these weights the amount of added cane sugar in the finished product was calculated.

TABLE 14.—*Composition of fruit.*^a

Serial number.	Description of sample.	Total solids.	Ash.	Total acids expressed as H ₂ SO ₄ .	Proteids (N × 6.25).	Sugars.		Polarizations.		
						Reducing sugar.	Cane sugar.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	°V.	°V.	°V.
20449	Apple (fall pippin)....	8.25	0.30	0.499		4.13	1.03	-2.2	-3.6	-1.8
20415	Blackberry	9.62	.60	.916	0.725	5.67		-1.1	-1.0	0
20418	Crab apple	14.34	.84	.705	.418	5.68	1.70	-.7	-3.0	-.8
20417	Grape (Ives seedling) ..	12.50	.75			6.11	.29	-1.6	-2.0	0
20425	Orange (Florida navel)	13.11	.61	.686	.985	4.13	3.33	+2.0	-2.5	-.8
20444	Pineapple	13.71	.50	.392	.056	8.12	3.11	+1.0	-3.2	-0

^aThe composition here given is not that of the original fruit, but of the pulped mass used in the preparation of jams.

TABLE 15.—*Composition of juice.*^a

Serial number.	Description of sample.	Total solids.	Ash.	Total acids expressed as H ₂ SO ₄ .	Proteids (N × 6.25).	Sugars.		Polarizations.		
						Reducing sugar.	Cane sugar.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	°V.	°V.	°V.
20400	Apple (fall pippin)....	7.95	0.47	0.627	0.543	4.00	1.18	-3.0	-4.6	-2.9
20396	Blackberry	8.54	.52	.978	.350	4.34	.00	-1.5	-1.6	-1.0
20403	Crab apple	5.62	.20	.372	.075	2.56	1.03	-1.0	-2.4	-1.1
20428	Grape (fox)	6.67	.49	1.686		2.79	.37	-.8	-1.3	-1.1
20401	Grape (Ives seedling) ..	8.83	.57	.902	.237	5.10	.89	-1.2	-2.4	-.6
20397	Huckleberry	16.33	.40	.454		11.21	.89	-3.2	-4.4	-.9
20430	Orange (Florida navel)	6.08	.36	.297	.581	1.52	2.29	+1.8	-1.3	0
20427	Peach	8.90	.45		.218		4.59	+4.0	-2.2	-.7
20431	Pear (Bartlett)	11.65	.45	.345	.087	5.87	1.18	-4.8	-6.4	-4.0
20429	Pineapple	13.27	.45	.588	.368	2.74	8.96	+8.4	-3.7	-1.1
20426	Pineapple-husk juice..	8.43	.77		.350		4.73	+4.1	-2.3	-.7
20399	Plum (damson)	12.72	.63		.431	4.86	.51	+2.0	+1.3	+2.4
20402	Plum (wild fox)	11.23	.64	1.576	.137	2.87	2.81	+1.4	-2.4	-1.8
20398	Mixed fruit	6.53	.32	.612	.150	2.68	.59	-1.0	-1.8	-.9

^aThe "juice" was prepared by boiling the fruit till soft, after the addition of sufficient water to prevent scorching, and straining through a jelly bag.

TABLE 16.—*Composition of jam.*

Serial number.	Description of sample.	Total solids.	Ash.	Total acids expressed as H ₂ SO ₄ .	Proteids (N × 6.25).	Sugars.				Polarizations.		
						Reducing sugar.	Cane sugar added.	Cane sugar found.	Cane sugar inverted.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	°V.	°V.	°V.
20446	Apple (fall pippin)....	63.22	0.20	0.282	0.175	25.52	51.31	29.11	43.22	+26.3	-13.0	+4.8
20414	Blackberry	55.42	.48	.851	.737	18.77	43.99	29.00	34.08	+24.6	-14.6	+1.6
20445	Grape (fox)	61.80	.19	.698	.200	50.06	54.21	3.70	92.96	-9.0	-14.0	+2.2
20416	Grape (Ives seedling) ...	56.64	.48	.744	.525	33.44	42.45	11.33	73.38	+3.5	-11.8	0
20443	Orange (Florida navel) .	80.52	.44	.433	.944	13.61	69.13	54.23	21.55	+55.9	-17.5	+2.0
20448	Pear (Bartlett)	61.52	.28	.163	.312	13.20	46.52	33.74	18.87	+32.3	-13.2	+1.0
20442	Pineapple	73.92	.30	.315	.312	14.05	60.20	46.40	22.90	+52.3	-10.3	+6.2
20421	Plum (damson)	50.43	.54	1.012	.525	28.29	37.75	9.70	74.42	+3.1	-10.0	+1.2
20423	Plum (wild fox)	62.10	.46	1.355	.212	28.78	47.86	23.26	53.43	+13.9	-17.5	0

TABLE 17.—*Composition of jelly.*

Serial number.	Description of sample.	Total solids.	Ash.	Total acids expressed as H ₂ SO ₄ .	Proteids (N × 6.25).	Sugars.				Polarizations.		
						Reducing sugars.	Cane sugar added.	Cane sugar found.	Cane sugar inverted.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
		<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	°V.	°V.	°V.
20408	Apple (fall pippin).....	59.18	0.22	0.279	0.175	20.78	51.76	33.04	36.17	+24.0	-20.6	-1.2
20405	Blackberry	59.63	.33	.475	.243	12.51	54.89	44.90	18.20	+47.0	-20.1	0
20410	Crab apple	63.28	.11	.171	.137	34.93	57.61	23.68	58.88	+13.0	-19.0	0
20405	Grape (Ives seedling) ...	63.66	.45	.524	.175	32.29	60.29	30.52	49.33	+22.3	-18.9	+ .2
20412	Huckleberry	63.02	.28	.245	.069	24.27	53.39	32.74	37.54	+24.1	-20.1	- .4
20435	Orange (Florida navel) .	68.56	.30	.171	.418	3.95	65.59	62.52	4.91	+61.3	-23.1	- .2
20437	Peach	69.98	.21	.245	.175	8.75	63.70	56.59	11.16	+53.4	-23.0	- .6
20434	Pear (Bartlett)	69.12	.34	.181	.156	6.58	63.09	58.46	7.33	+52.7	-26.2	-1.8
20436	Pineapple	80.28	.43	.328	.387	22.13	72.98	56.70	28.45	+50.4	-26.1	0
20433	Pineapple husk.....	76.34	.73	.352	.350	7.40	70.22	65.22	7.12	+63.7	-24.3	- .6
20404	Plum (damson)	45.56	.68	1.127	.350	19.18	38.00	22.67	40.38	+17.8	-12.8	0
20409	Plum (wild fox).....	54.49	.40	1.029	.138	24.00	48.05	25.48	46.97	+16.7	-17.8	0
20411	Plum (wild fox), boiled down	73.01	.65	1.529	.175	44.22	64.66	22.37	66.18	+ 7.6	-22.6	- .6
20407	Mixed fruit.....	66.58	.21	.367	.069	39.70	59.72	24.22	40.38	+14.8	-17.9	+2.2

The analyses of the fruits and juices in these tables are not comparable, owing to the fact that the amount of water added in their preparation was varied in order to obtain the desired product. Regarding the content of cane sugar, the analyses are in accord with those already given.

The inversion of cane sugar during the process of preparation, as shown in the analyses of jams and jellies, is of particular interest. The amount of inversion that takes place varies in general with the length of time of heating and with the content of acid, although other factors have a material influence. It is well known that the different fruit acids vary widely in their power of inverting cane sugar, and many of the variations are undoubtedly due to this cause. Apple jam, with an acidity of 0.282 per cent, shows a much larger amount of cane sugar inverted than blackberry, orange, and pineapple jams, which have an acidity of 0.851 per cent, 0.433 per cent, and 0.315 per cent, respectively, while grape jam, with an acidity of 0.698 per cent, shows almost complete inversion of the cane sugar. Wild fox plum jam, with an acidity of 1.355 per cent, shows but a slightly larger amount of sugar inverted than the apple jam. The amount of sugar inverted in the jellies is much less than in the corresponding jams, owing to the fact that they were heated for a much shorter time. The amount of inversion was very small in case of the orange, pineapple husk, and peach. In all of these cases the acidity is low. The Ives grape, wild fox plum, and mixed fruit jellies all

show a high percentage of inversion as well as a high acidity. The crabapple jelly is a striking exception. Its acidity is very low, yet the amount of inversion is high.

The invert polarizations of the jams at 86° are peculiar in that they are strongly positive, with two exceptions, while the same readings made on the fruits, juices, and jellies are either zero or negative, with the exception of plum juice and mixed fruit jelly. The cause of this change has not been worked out, but it is probable that some of the insoluble matters of the fruit have been changed to soluble, optically active bodies by the continued heating of the jams.

Tables 16 and 17 show to very good advantage the relative amounts of protein present in the jams and the jellies. The average content of protein for the eight jams given is 0.468 per cent, while for the corresponding jellies the protein content is only 0.230 per cent. In no case is the content of protein sufficiently high in the jelly to interfere with the detection of added gelatin by the simple method of determining the nitrogen content.

COMMERCIAL FRUIT PRODUCTS.

The fruit products examined have been divided into jams, jellies, canned fruits and miscellaneous fruit products, brandied fruits, fruit butters, and marmalades. The meaning of the term "jelly" is well understood, but there is considerable difference of opinion regarding the definition of jams and marmalades. Undoubtedly the usage varies in different parts of the country and with different manufacturers. Products in every respect similar with trade names and secret formulæ for their preparation, are often represented as entirely distinct products. But for the sake of convenience in comparison and discussion in the following tables jams include marmalades and fruit products in which the pulped fruit is reduced to the consistency of a jam. The term "brandied fruits" needs no explanation. Fruit butter is the boiled-down fruit juice containing some of the pulp, but as a rule no sugar is added. In the preparation of this product the methods may vary widely in different sections of the country, depending upon the kinds of fruit available. In case sour apples are employed, cane sugar may be added, while if sweet apples are used an addition of cane sugar will not be necessary.

JAMS.

Discussion.—Analyses of 96 samples of jams were made. Of this number, 86 were commercial products and 10 were prepared in the laboratory. Of the commercial jams, 18 samples, less than 20 per cent, contained no glucose, 53 contained glucose, but were not so labeled, and the remaining 15 were purchased as compound goods. The jams are arranged according to the above classification in three sets of tables which follow the discussion of this subject.

The actual percentage of solids in these products has but little value in the determination of purity, for it varies within wide limits—from a maximum of 82.46 per cent to a minimum of 50.43 per cent, a difference of 32 per cent. The difference, however, between the total sugar and the soluble solids in glucose goods may serve as a valuable indication of the amount of dextrin present, and thus the approximate amount of glucose which has been added, as the amount of sugar-free soluble solids is fairly constant in pure products.

The percentage of ash is of little value in judging purity, although the glucose products as a rule have a high ash. Many of the pure-fruit products also have a high ash. The relation between the total ash and the alkalinity of the ash, calculated as potassium carbonate (K_2CO_3), is of marked value in the pure-fruit goods, and differs materially from that of the ash of goods mixed with glucose. The ash of fruits is largely potassium carbonate, as is shown in the following table:

TABLE 18.—*Ash of fruits.*

Fruit.	Pure ash.	K ₂ O.	Na ₂ O.	CaO.	P ₂ O ₅ .	SO ₃ .	Cl.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Apple ^a	0.264	55.21	11.69	4.79	12.83	4.62	0.83
Apricots ^b	.508	59.36	10.26	3.17	13.09	2.63	.45
Banana ^c	1.078	63.06	2.34	.86	1.62	2.32	26.93
Cherries ^d	.440	57.67	6.80	4.20	15.11	5.83	1.83
Figs ^e	.682	57.16	2.38	10.90	12.76	3.90	2.05
Grapes ^f	.500	50.95	6.32	4.96	21.27	4.28	1.54
Lemons ^g	.526	48.26	1.76	24.87	11.09	2.84	.39
Oranges ^g	.432	48.94	2.50	22.71	12.37	5.25	.92
Prunes ^h	.486	63.83	2.65	4.66	14.08	2.68	.34

^a Tolman, Calif. Expt. Sta. Report, 1900, p. 146.

^b G. E. Colby, Calif. Expt. Sta. Report, 1894, p. 266.

^c Ibid., p. 279.

^d Ibid., 1896, p. 179.

^e Ibid., 1894, p. 233.

^f F. T. Bioletti, Calif. Expt. Sta. Report, 1894, p. 274.

^g G. E. Colby, Calif. Expt. Sta. Report, 1890, p. 113.

^h Ibid., 1892, p. 266.

It will be seen that there are only small percentages of sulphates and chlorids in the total ash. The chlorid in nearly all is especially low. The banana ash is an exception, being practically all potassium chlorid. In the quantity of substances taken in the ordinary determination of ash of fruit products the sulphates and chlorids in products containing no glucose will amount only to traces, except as has been noted in the banana.

An appreciable addition of glucose, the ash of which is largely composed of sulphates and chlorids, will greatly change the relation between the alkaline carbonates and the sulphates and chlorids. If this table of fruit ashes be compared with that of glucose ashes (Table 5, p. 33) the great difference will be noted. If a still more complete examination of the ash is made a more striking difference might be shown in the proportion of the different bases. The bases in fruit ash are very largely potash with only small amounts of soda and lime, while in the glucose ash soda and lime are greatly in excess and only traces of pot-

ash will be found. Still, exceptions to this rule must not be overlooked. In several analyses of ash of apple considerable amounts of soda have been shown to be present. Also, in some fruits, such as lemons and oranges, the percentage of lime is much larger than is the case with most other fruits. The ash of a pure fruit product, however, should contain not less than 40 per cent of potash.

By consulting the tables of analyses it will be noticed that the ash of the glucose products varies exceedingly, according to the grade of glucose employed and the method of its preparation. The sulphates in some cases constitute almost the entire ash, while in other cases only a very small part of the ash is sulphate. The average percentage of ash of jams not containing glucose was 0.319, while the average alkalinity was 0.262. In the compound jams the average ash was 0.590 per cent and the alkalinity 0.285 per cent. These proportions, however, will not always hold good. For example, sample No. 19887, which is very largely glucose, has 0.387 per cent of ash and only small amounts of sulphates and chlorids. This sample evidently contains a better grade of glucose than that used in sample No. 19865, which has 0.648 per cent of ash and 0.546 per cent of sulphates. The dextrin and sugar in the sample of glucose last mentioned are present in about equal proportions. An ash of this sort indicates that there can be very little if any fruit present.

There is little value in a comparison of jams made from different fruits by taking as a basis the amount of any constituent present, as the variation is too great. The goods of one manufacturer, however, may be compared with considerable value sometimes, as is seen by consulting Table 31, prepared to show the probability of the use of a gelatinizing agent to produce a uniform product from different kinds of fruits. It is well known that some fruits jelly readily, while others do not, and with many fruits it is necessary to have a high content of solids in order to get the required consistency. By the use of apple as a basis it is possible to produce the desired consistency with a lower percentage of solids.

The relation between cane sugar and reducing sugar in jellies or jams is of little value, as the amount of hydrolysis caused by the fruit acids in the process of preparation is an exceedingly variable quantity, changing with the amount of boiling and the quantity and kind of acid present. Data of considerable value may be obtained by a consideration of the total sugar present and the invert polarization. The following table was made by calculating all the reducing sugar as cane sugar (reducing sugar $\times .95$ = cane sugar), and adding it to the cane sugar found, and from the total cane sugar, calculating the invert reading at the temperature at which the polarizations of the sample were made. It is easily seen that if the fruit product had contained no sugar besides the sugar of the fruit and pure cane sugar the observed polarization and the calculated polarization would be very close

together, while in the presence of glucose the calculated polarization to the left on the sugar scale is larger than the actual reading, the difference depending on the amount of glucose present. The difference between the calculated reading and the actual reading is marked plus (+) when the former is the greater and minus (—) when the actual reading is greater than that calculated. If all the sugar in the fruits were present as invert and cane sugar it would be possible to make quite exact calculations, but, as has been shown in Tables 14 and 16, there is often a small excess of either levulose or dextrose. This is not sufficiently marked, however, to make any serious difference, especially as in nearly all the fruits examined the levulose is in excess, which lessens the difference mentioned above. A small difference of one or two degrees either one way or the other should be disregarded, as it may be caused by the slight excess of levulose or dextrose in the fruit. The following table illustrates this point very plainly:

TABLE 19.

POLARIZATION OF JAMS NOT CONTAINING GLUCOSE.

Serial number.	Per cent total sugar as cane sugar.	Temperature in degrees C.	Invert polarization.	Calculated invert polarization.	Difference.
20446	53.36	18	-13.00	-18.7	+5.7
20183	62.95	20	-20.80	-21.4	+ .6
20414	46.77	18	-14.60	-16.4	+1.8
20175	51.47	20	-18.00	-17.5	- .5
21081	60.97	20	-20.00	-20.7	+ .7
21082	63.01	21	-12.40	-21.1	+8.7
20163	67.16	21	-17.00	-20.4	+3.4
20416	43.06	18	-11.80	-15	+3.2
20445	51.27	18	-14.00	-17.9	+3.9
21083	76.61	21	-25.20	-25.6	+ .4
20245	62.65	21	-19.80	-20.9	1.1
20025	59.33	20	-19.00	-20.1	1.1
20196	57.85	21	-18.80	-19.4	+ .6
20443	67.16	18	-17.50	-23.5	+4.0
20589	66.58	20	-23.00	-22.6	- .4
20968	60.05	20	-18.80	-20.4	+1.6
20448	46.28	18	-13.20	-16.2	+3.0
20181	57.82	20	-19.40	-19.6	+ .2
20967	59.54	20	-19.80	-20.2	+ .4
20179	60.75	20	-18.30	-20.6	+2.3
20421	36.58	18	-10.00	-12.8	+2.8
20423	50.60	18	-17.50	-17.71	+ .2
20591	55.38	20	-18.05	-18.8	+ .75
20182	66.72	22	-22.80	-22	- .8
20590	69.14	20	-21.50	-22.5	+2.0

POLARIZATION OF JAMS CONTAINING GLUCOSE.

20166	57.45	20	- 7.70	-19.5	+11.8
20165	59.08	20	- 3.80	-20	+16.2
21021	66.16	20	- 1.60	-22.5	+20.9

The pure fruit jams prepared in the laboratory for comparison and those commercial products that contained no glucose are arranged first. The difference between the calculated and determined polarization is, as a rule, less than 3° . Among the jams of known origin there are three exceptions to this, the maximum being $+5.7$ on apple jam 20446. The commercial goods which would ordinarily be passed as free from glucose afford one exception, and this is probably due to the addition of a small amount of commercial dextrose which contained no dextrin, or possibly to the use of cane sugar adulterated with dextrose. There is said to be on the market at present cane sugar mixed with crystallized dextrose, often as high as 20 per cent of the latter being present. In some cases this is also mixed with saccharin in order to secure the desired sweetening power. The value of this calculation is brought out very plainly when these results are compared with those obtained with the three samples of jam which have a minus polarization but which contain a small amount of glucose, viz, 20166, 20165, and 21021, in which the minimum difference between the calculated and determined polarizations is $+11.8$ and the maximum $+20.9$.

In pure jellies the agreement between invert polarization and that calculated as indicated above is even more striking than in the case of jams, the maximum difference being 3.7° while as a rule the difference is not more than 1.5° . The five commercial jellies containing small amounts of glucose show a minimum difference of $+11.86^{\circ}$

TABLE 20.

POLARIZATION OF JELLIES MADE IN LABORATORY.

Serial number.	Per cent total sugar as cane sugar.	Temperature in degrees C.	Invert polarization.	Calculated invert polarization.	Difference.	Polarization at 86° .
20408	52.79	18	-20.6	-18.48	- 2.2	-1.2
20405	55.81	18	-20.1	-19.5	- .6	0.0
20410	56.85	18	-19.0	-19.90	+ .9	0.0
20405	61.19	18	-18.9	-21.40	+ 2.5	+ .2
20412	55.80	18	-20.1	-19.5	- .6	- .4
20435	66.27	18	-23.1	-23.20	+ .1	- .2
20437	64.90	18	-23.0	-22.7	- .3	- .6
20434	64.71	18	-26.2	-22.6	- 3.4	-1.8
20436	77.73	18	-26.1	-27.2	+ 1.1	0.0
20433	72.25	18	-24.3	-25.2	+ .9	-0.6
20404	40.91	18	-12.8	-14.3	+ 1.5	0.0
20409	48.28	18	-17.8	-16.8	- 1.0	0.0
20411	64.41	18	-22.6	-22.5	- .1	-0.6
20407	61.92	18	-17.9	-21.6	+ 2.7	+2.2

TABLE 20—Continued.

POLARIZATION OF COMMERCIAL JELLIES CONTAINING NO GLUCOSE.

Serial number.	Per cent total sugar as cane sugar.	Temperature in degrees C.	Invert polarization.	Calculated invert polarization.	Difference.	Polarization at 86°.
20023	57.83	20	-17.9	-19.8	+ 1.9	
20226	60.47	20	-22.3	-20.5	- 1.8	
21062	53.75	20	-20.6	-18.2	- 2.4	
19890	56.35	20	-18.1	-19.1	+ 1.0	
20024	57.26	20	-18.7	-19.4	+ .7	
20169	62.99	20	-21.3	-21.3	0.0	
20170	62.93	20	-20.0	-21.3	+ 1.3	
20180	66.95	20	-22.3	-22.7	+ .4	
20210	61.80	20	-22.0	-21.0	- 1.0	
20214	52.50	20	-18.2	-17.9	- .3	
20184	61.63	20	-19.2	-20.9	+ 1.7	
20189	64.53	20	-21.9	-21.9	0.0	
20246	73.31	20	-24.8	-24.9	+ .1	
21084	73.39	20	-24.1	-24.9	+ .8	
20173	58.79	20	-19.8	-19.9	+ .1	
20185	60.93	20	- 8.8	-20.7	+11.9	
20188	67.31	20	-20.8	-22.8	+ 2.0	
20172	61.52	20	-17.6	-20.9	+ 3.3	
20186	60.34	20	-16.8	-20.5	+ 3.7	

POLARIZATION OF JELLIES CONTAINING GLUCOSE.

20213	51.55	20	- 5.35	-17.55	+12.20	
20966	64.59	20	- 4.00	-21.95	+17.95	
20212	49.57	20	- 5.00	-16.86	+11.86	
20211	51.76	20	- 4.00	-17.60	+13.60	
20185	60.93	20	- 8.80	-20.70	+12.90	

Sample 20185 is especially to the point. It has an invert reading of -8.80 and no dextrin was found by fermentation, but the difference of +12.90 indicates the presence of a dextrorotatory sugar, in all probability dextrose. It is in such cases that the method of comparison shows the great value of polarization after inversion for the detection of a small amount of added dextrose which may not contain enough dextrin to be detected by the fermentation process.

The polarization after fermentation with yeast, by which all the sugars are removed, is approximately zero and does not vary more than $\pm 0.3^\circ$ in the case of products which contain no glucose, but in the glucose products the right-handed polarization of the dextrin is unchanged. Great care must be used, however, to secure complete fermentation or very erroneous results will be obtained. Dextrose is more easily fermented than levulose, so that a zero reading could be obtained even though a considerable amount of dextrin were present, the left-handed reading of the levulose neutralizing the right-

handed reading of the dextrin. The presence of preservatives interferes to a considerable extent and in some cases prevents complete fermentation. As a result left-handed readings were obtained which showed that considerable of the levulose was still unfermented. It is always well to test for reducing sugars even if a zero reading has been obtained for the reason mentioned above.

The percentage of nitrogenous material in the jams is quite variable—much more so than in the jellies—on account of the pulp present in the former, which contains a large part of the nitrogenized bodies in an insoluble condition, as can be seen by comparing the protein of juices and whole fruits in Tables 14 and 15. The percentage of protein in jam, therefore, will depend largely upon the amount of fruit pulp used and is thus an indication of the amount of fruit employed, which in some of the jams is evidently rather small. The protein contained in compound jams and jellies (see Tables 26 and 33) is nearly the same, which is quite different from the condition found in the fruit jams and jellies which contained no glucose. The other constituents of the compound goods also vary little, showing that there is little difference in the process of making them or the ingredients used.

The high protein content of sample 20175 perhaps needs some explanation. The currants used in the preparation of this jam were hard and tough and looked as though they had been dried before being used in the preparation of the jam. As a result the insoluble matter is very high (6.32 per cent), as are also the proteids (1.410 per cent), the ash (0.841 per cent), and the acid (1.117 per cent).

There is little that can be said about the acid content except that certain fruits, such as currants and plums, have a high content of acid as a rule, and jellies and jams made from them should also have a high acidity. For this reason the acid content may sometimes be of value as evidence that there has been some substitution of other fruits for those mentioned.

Jams containing no glucose.—Of the 18 commercial samples in the table, about one-third contained preservatives and 3 were artificially colored with coal-tar dyes.

TABLE 21.—*Description of jams not containing glucose.*

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Apple.</i>		
20446	Made in laboratory		
	<i>Apricot.</i>		
20183	J. Keiller, Dundee	N. W. Burchell, Washington, D. C.	
	<i>Blackberry.</i>		
20414	Made in laboratory		

TABLE 21.—Description of jams not containing glucose—Continued.

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Currant.</i>		
20175	Mrs. J. T. McCready, Buffalo, N. Y.	C. C. Bryan, Washington, D. C.	Bar le due.
21081		S. W. Clark & Son, 626 Canal street, New Orleans, La.	
21082		do	
	<i>Fig.</i>		
20241	Barataria Canning Co., Biloxi, Miss.	Park & Tilford, New York City...	
	<i>Grape.</i>		
20416	Made in laboratory		
20445	do		
	<i>Grape fruit.</i>		
20163	Bishop & Co., Los Angeles, Cal ...	C. C. Bryan, Washington, D. C.	Grape fruitate.
	<i>Guava.</i>		
21083	J. Cernel, Ormonde, Fla	S. W. Clark & Son, 626 Canal street, New Orleans, La.	Marmalade.
20245	Bishop & Co., Los Angeles, Cal ...	Park & Tilford, New York City...	Do.
	<i>Orange.</i>		
20025	J. Buchanan, Ltd., Glasgow, Scotland.	R. H. Macy & Co., New York City.	Homemade marmalade.
20196		G. G. Cornwell & Son, Washington, D. C.	Do.
20443	Made in laboratory		
20589	J. Keiller & Son, Dundee	Jackson & Co., 114 West 23d street, New York City.	
20968	Crosse & Blackwell, London.....	A. M. & J. Solari, Royal and Canal streets, New Orleans, La.	Orange marmalade.
	<i>Pear.</i>		
20448	Made in laboratory		
	<i>Peach.</i>		
20181	Oneida Community, Ltd., Kenwood, N. Y.	N. W. Burchell, Washington, D. C.	Peach jam.
20967	Crosse & Blackwell, London.....	A. M. & J. Solari, New Orleans, La.	
	<i>Pineapple.</i>		
20442	Made in laboratory		
	<i>Plum.</i>		
20179	Crosse & Blackwell, London.....	N. W. Burchell, Washington, D. C.	Green gage.
20421	Made in laboratory		
20423	do		
20591	J. Keiller & Son, Dundee	Jackson & Co., 114 West 23d street, New York City.	Damson.
	<i>Strawberry.</i>		
20182	Oneida Community, Ltd., Kenwood, N. Y.	N. W. Burchell, Washington, D. C.	Strawberry jam.
20590	J. Keiller & Son, Dundee.	Jackson & Co., 114 West 23d street, New York City.	

TABLE 22.—Composition of jams not containing glucose.

Serial number.	Description.	Percent total solids.	Per cent insoluble solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Per cent alcohol precipitate.	Per cent sugar-free soluble solids.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as potassium sulphate.	Chlorids as sodium chloride.	Preservatives.	Foreign coloring matter.
						Per cent reducing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature.									
20446	Apple	63.22		0.175	0.282	25.52	29.11	54.63	+26.30	-13.00	18			8.59	0.200					
20483	Apricot	70.15	1.72	.494	.407	38.96	26.00	64.96	+14.05	-20.80	20	None.	1.144	3.37	.348	0.295	Trace.	Trace.		
20414	Blackberry	55.42		.737	.851	18.77	29.00	47.77	+24.60	-14.60	18			7.65	.480					
20775	Current	66.32	6.32	1.410	1.117	52.45	1.64	54.09	-15.80	-18.00	20	None.	.920	5.96	.841	.603	0.098	0.014		
21081	do	70.56	.98	.175	.431	58.52	5.37	61.89	-12.80	-20.00	20	None.		5.69	.159	.164	Trace.	Trace.	Benzoic acid.	Magenta
21082	do	71.30		.325	.475	51.04	14.53	65.57	+7.00	-12.40	20	None.		5.73	.123	.106	Trace.	Trace.	Salicylic acid.	
20241	Fig	69.89					45.92		+44.80	-20.10	20				.193	.184				
20416	Grape	56.64		.525	.744	33.44	11.33	44.77	+3.50	-11.80	18			11.87	.480					
20445	do	61.80		.200	.698	50.06	3.70	53.76	-9.00	-14.00	18			8.04	.190					
20163	Grape-fruit	69.20	1.25	.350	.387	27.00	35.51	62.51	+30.40	-17.00	20	None.		5.44	.334	.301	.043	.008	Benzoic acid.	
21083	Guava	82.46	1.04	.175	.299	25.14	52.73	77.87	+45.20	-25.20	21	None.		3.58	.301	.261	Trace.	Trace.		Red coal tar.
20245	do	70.97	3.30	.388	.446	43.56	21.27	64.83	+8.60	-19.80	21	None.		2.85	.332	.313	.008	.009		
20025	Orange	65.44	.09	.212	.490	51.14	10.75	59.33	-4.60	-19.00	20	None.	.540	3.46	.143	.100	Trace.	Trace.		
20196	do	63.90	1.03	.175	.206	45.18	14.91	57.85	+1.10	-18.80	21	None.		2.78	.138	.156	Trace.	Trace.		
20443	do	80.52		.941	.433	13.61	54.23	67.84	+55.90	-17.50	18			12.68	.440					
20589	do	72.76	.89	.206	.485	33.41	34.85	68.26	+23.70	-23.00	20	None.	1.764		.207	.206	Trace.	Trace.		
20968	do	67.99	1.32	.244	.519	61.02	2.09	63.11	-16.00	-18.80	20	None.	1.418	3.56	.292	.259	.028	Trace.		
20484	Pear	61.52		.312	.163	13.20	33.74	46.94	+32.30	-13.20	18			14.58	.280					
20181	Peach	65.65	1.68	.388	.500	36.48	23.16	59.64	+11.40	-19.40	20	None.		4.33	.391	.286	Trace.	Trace.		
20967	do	67.33	1.08	.350	.259	46.78	15.08	61.86	+45	-19.80	20		1.494	4.39	.275	.199	Trace.	Trace.		

TABLE 22.—Composition of jams not containing glucose—Continued.

Serial number.	Description.	Per cent total solids.	Per cent insoluble solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free soluble solids.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ O ₈ .	Sulphates as potassium sulphate.	Chlorids as sodium chloride.	Preservatives.	Foreign coloring matter.	
						Per cent reducing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature.											
20442	Pineapple	73.92		.312	.315	14.05	46.40	60.45	+52.30	-10.30	18				13.47	.300						
20179	Plum	70.19	.96	.281	.407	50.34	12.91	63.25	-1.00	-18.30	20	None.	1.090	5.98	6.94	.263	.210	Trace.	Trace.			
20421	do	50.43		.525	1.012	28.29	9.70	37.99	+3.10	-10.00	18				12.44	.540						
20423	do	62.10		.212	1.355	28.78	23.26	53.04	+13.90	-17.50	18				10.06	.460						
20591	do	64.78	2.57	.456	.804	45.76	11.90	57.66	-2.20	-18.05	20	None.	1.630	4.55	7.12	.320	.270	Trace.	Trace.	Salicylic acid.		
20182	Strawberry	75.83	4.59	.769	.480	37.15	31.43	68.58	+19.00	-22.80	22	None.		2.66	7.25	.422	.318	.046	.010	do		
20590	do	69.16	1.90	.419	.529	38.50	22.57	61.07	+8.75	-21.50	20	None.	.902	6.19	8.09	.337	.222	Trace.	Trace.	do		
	Average	65.98	1.92	.430	.536	36.41	22.15					0.007	1.119	4.40	7.71	.319	.262	.016	.002			
	Maximum	82.46	6.32	1.410	1.355	61.02	54.23					.070	1.764	6.19	14.58	.841	.603	.098	.014			
	Minimum	50.43	.09	.175	.163	13.20	.30					None.	.092	2.66	3.55	.143	.100	Trace.	Trace.			

^a See correspondence with manufacturers, p. 103.

Jams containing glucose, but not so labeled.—The table of products coming under this head includes goods of nearly all grades from the poorest to the best. The nitrogen content is variable, depending somewhat upon the percentage of fruit used. The percentage of insoluble solids and of protein vary directly with each other. Out of 53 samples 28 were found to be colored, the reason being that the pulp does not retain its color as well as the juice does, and, further, the use of coloring materials permits the employment of a poor grade of fruit or the substitution of a cheaper fruit.

TABLE 23.—*Description of jams containing glucose, but not so labeled.*

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Apricot.</i>		
20206	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, apricot jam.
	<i>Blackberry.</i>		
20147	Williams Bros. & Charbonneau, Detroit.	Henry Barge, New York	Prepared with best granulated sugar, guaranteed absolutely pure.
F 651	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill	Quince; Genesee fresh fruit preserves.
	<i>Cherry.</i>		
19880	Milford Manufacturing Co., Chicago, Ill.	Siegel-Cooper Co., New York.....	Red cherry jam.
19883	do	do	Do.
19957	Alden & Nicholson, Rochester, N. Y.	H. Punchard & Sons, New York ..	Fresh fruit jam—cherry.
19960	Geo. K. McMechen & Son, Wheeling, W. Va.	do	Aunt Dinal brand, homemade fresh fruit jam.
20022	R. H. Macy & Co., New York City.	R. H. Macy & Co., New York.....	Lily White brand, strictly pure fruit.
20027	Anderson Preserving Co., Camden, N. J.	do	Cherry jam (in tin).
20159	Francis H. Leggett & Co., New York.	Henry Barge, New York	Fruit jam.
20177	Flacus Bros., Wheeling, W. Va..	N. W. Burchell, Washington, D. C.	
20199	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, cherry jam.
20218	P. J. Ritter Conserve Co., Philadelphia, Pa.	J. C. Ergood Co., Washington, D. C.	XX brand, fresh fruit and granulated sugar.
	<i>Currant.</i>		
19881	Milford Manufacturing Co., Chicago, Ill.	Siegel-Cooper Co., New York.....	Red currant jam.
19884	do	do	Do.
20026	Anderson Preserving Co., Camden, N. J.	R. H. Macy & Co., New York.....	Currant jam (in tin).
20166	A. Cairns, Paisley, Scotland	C. C. Bryan, Washington, D. C. ...	Finest Scotch preserves, whole fruit, special quality.

TABLE 23.—*Description of jams containing glucose, but not so labeled*—Continued.

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Currant</i> —Continued.		
20204	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, currant jam.
20207	do.....	do.....	Do.
20220	P. J. Ritter Conserve Co., Philadelphia, Pa.	J. C. Ergood Co., Washington, D. C.	Pomona, pure fruit, currants.
21022	J. Moir, London.....	A. L. Buhler Co., Decatur street, New Orleans, La.	Red currants.
F 655	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill.....	Currant; Genesee fresh fruit preserves.
	<i>Gooseberry.</i>		
19878	Anderson Preserving Co., Camden, N. J.	Siegel-Cooper Co., New York.....	Gooseberry jam (in tin).
20201	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, gooseberry jam.
	<i>Grape.</i>		
19877	Anderson Preserving Co., Camden, N. J.	Siegel-Cooper Co., New York.....	Grape jam (in tin).
	<i>Orange.</i>		
20198	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, orange marmalade.
20584	W. P. Hartley, Liverpool.....	R. M. Delapenha & Co., 81 Murray street, New York.	Made from Seville oranges.
	<i>Peach.</i>		
19876	Anderson Preserving Co., Camden, N. J.	Siegel-Cooper Co., New York.....	Peach jam (in tin).
20158	Francis H. Leggett & Co., New York City.	Henry Barge, New York.....	Fruit jam.
	<i>Pear.</i>		
20187	Geo. K. McMechen & Son, Wheeling, W. Va.	N. W. Burchell, Washington, D. C.	Old Virginia brand.
20197	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, pear jam.
	<i>Plum.</i>		
20021	R. H. Macy & Co., New York City..	R. H. Macy & Co., New York.....	Green gage.
20028	Anderson Preserving Co., Camden, N. J.	do.....	Plum jam (in tin).
20148	Williams Bros. & Charbonneau, Detroit.	Henry Barge, New York.....	Prepared with best granulated sugar and guaranteed pure.
20200	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, plum jam.
F 652	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill.....	Green gage; Genesee fresh fruit preserves.
	<i>Quince.</i>		
19967	Williams Bros. & Charbonneau, Detroit.	H. Punchard & Son., New York...	Prepared with best granulated sugar; guaranteed absolutely pure.
20202	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, quince jam.
F 657	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill.....	Quince; Genesee fresh fruit preserves.

TABLE 23.—*Description of jams containing glucose, but not so labeled—Continued.*

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
<i>Raspberry.</i>			
19963	Franklin Preserving Co., New York.	H. Punchard & Son, New York....	High grade goods.
19879	Milford Preserving Co., Chicago..	Seigel-Cooper Co., New York	
19959	Alden & Nicholson, Rochester, N. Y.	H. Punchard & Son, New York....	Highest grade fruit.
19961	Geo. K. McMechen & Son, Wheeling, W. Va.	do	Aunt Dinah brand, homemade fresh fruit jam.
19966	Williams Bros. & Charbonneau, Detroit.	do	Prepared with best granulated sugar, guaranteed absolutely pure.
20190	Alden & Nicholson, Rochester, N. Y.	N. W. Burchell, Washington, D. C.	Raspberry jam.
20203	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit.
20585	W. P. Hartley, Liverpool.....	R. M. Delapenha & Co., 81 Murray street, New York.	Do.
19958	Alden & Nicholson, Rochester, N. Y.	H. Punchard & Son, New York....	Highest grade fruit.
F 654	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill	Raspberry; Genesee fresh fruit preserves.
<i>Strawberry.</i>			
19962	Franklin Preserving Co., New York.	H. Punchard & Son, New York....	Highest grade fruit jam.
19964	Alden & Nicholson, Rochester, N. Y.	do	Highest grade fruit.
19988	Hazel Pure Food Co.....	Siegel-Cooper Co., New York	Hazel brand.
20146	Williams Bros. & Charbonneau, Detroit.	Henry Barge, New York.....	Prepared with best granulated sugar, guaranteed absolutely pure.
20165	Lutz & Schramm	C. C. Bryan, Washington, D. C.....	
20167	A. Cairns, Paisley, Scotland.....	do	Finest whole fruit, special quality.
20205	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	Fresh fruit, strawberry jam.
21021	J. Moir, London	R. H. Macy & Co., New York.....	Strawberry jam.
F 653	Batavia Preserving Co., Batavia, N. Y.	Boston Store, Chicago, Ill	Strawberry; Genesee fresh fruit preserves.

TABLE 24.—Composition of jams containing glucose, but not so labeled.

Serial number.	Description.	Per cent total solids.	Per cent insoluble solids.	Per cent of protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Approximate per cent glucose (dextrin×3).	Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as per cent potassium sulphate.	Chlorids as per cent sodium chlorid.	Starch.	Preservatives.	Foreign coloring matter.
						Per cent reducing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, degrees C.											
20206	Apricot	70.10		0.594			25.32		+ 46.4	+ 12.6	21	13.65	4.55			0.387	0.299				Benzoic acid.	
20147	Blackberry ^a	69.69	1.41	.281	0.461	32.96	1.12	34.08	+107.0	+105.5	20	72.24	24.08		35.61	.458	.344	0.076	0.016			
F 651	do. ^b	74.95				34.72	20.53	55.25	+ 68.2	+ 41.2	21	(°)	(°)		19.70	.447	.273			Present.	Benzoic acid.	Red coal tar.
19880	Cherry	69.60				12.67			+ 76.8	+ 60.0	20										Benzoic acid.	Do.
19883	do.	71.35		.319		11.05			+ 74.8	+ 59.0	20	34.92	11.64			.409	.298				Salicylic acid.	Azo red.
19957	do. ^a	69.59			.333	2.86			+ 68.8	+ 65.0	20					.578	.226					
19960	do.	68.58	.38	.281	.353	28.00	18.95	46.95	+ 78.6	+ 53.2	20	48.60	16.20		21.24	.454	.286	.201	.003	Present.	Benzoic acid.	
20022	do.	63.67	2.03	.562	.558	35.14	11.54	46.68	+ 55.0	+ 39.6	21	30.72	10.24		16.99	.391	.311	.077	.010			Red coal tar.
20027	do.	71.82			.380	11.59			+ 84.9	+ 68.5	19.5					.475	.386				Benzoic acid.	Do.
20159	do.	68.60	1.61	.594	.372	37.12	22.03	59.15	+ 31.1	+ 1.8	22	12.87	4.29		9.45	.395	.324	.039	Trace.			
20177	do.	75.99		.525		8.54			+113.4	+102.0	20	59.10	19.70			.588	.375				Saccharin	
20199	do.	68.82	2.09	.563	.485	38.06	21.20	59.26	+ 25.8	+ 2.6	20	9.72	3.24			.353	.231	.043	.006		Benzoic acid.	
20218	do. ^a	75.32		.806		11.20			+ 58.6	+ 43.6		39.66	13.22			.526	.407				do.	Do.
19881	Current	72.91			.457	10.24			+ 79.20	+ 65.40	19.5					.410	.294					Do.
19884	do.	71.00	1.39	.388	.470	33.92	12.39	46.31	+ 81.00	+ 64.4	20	49.89	16.63		24.69	.410	.330	.093	.011			Azo red.
20026	do. ^a	69.99				7.64			+ 77.20	+ 67.1	21					.472	.275					Red coal tar.

20166	do.....	68.29	3.12	.294	1.039	54.21	5.93	60.14 +	.25	—	7.70	20	6.18	2.06		8.15	.315	.201	.058	.002		Salicylic acid. ^a	Do. Coch i-neal.
20204	do.....	63.46	1.77	.494	.558	36.10	10.22	46.32 +	50.00 +	36.30	20	31.87	10.59		17.14	.433	.311	.072	.030		Benzoic acid.	Do.	
20207	do.....	62.38	3.43	.738	.676	37.96	9.33	47.29 +	26.60 +	14.1	20	23.91	7.97		15.09	.456	.311	.054	.012		Present.	Red coal tar.	
20220	do. ^a	65.14	1.48	.419	.407	32.48	4.03	36.51 +	94.40 +	89.0	20	63.03	21.01		28.63	.565	.270	.279	.018		Abundant.	Do.	
21022	do.....	73.30	2.83	.806	.647	52.96	10.68	63.64 +	15.40 +	1.2	22	13.41	4.47		9.66	.453	.286	Trace.	Trace.		Benzoic acid.	Do.	
F 655	Current.....	71.32				41.60	12.47	56.07 +	62.8 +	46.4	21	(^c)	(^c)		15.25	.446	.246				Salicylic acid. ^a	Do.	
19878	Gooseberry ^a	71.02					4.36		+ 71.7 +	65.9	19.5					.493	.322				Salicylic acid. ^a	Do.	
20201	do.....	65.52		.456			5.84		+ 24.0 +	16.2	20	21.0	7.00			.387	.311				Benzoic acid.	Do.	
19877	Grape ^a	73.46					11.76		+ 81.6 +	65.9	21					.397	.229				Benzoic acid.	Do.	
20198	Orange.....	68.97	1.06	.312	.319	25.25	27.61	52.86 +	64.60 +	27.5	20	31.53	10.51		16.11	.373	.345	Trace.	.019		Benzoic acid.	Do.	
20584	do.....	71.81	1.22	.206	.559	36.66	25.75	62.41 +	37.30 +	2.80	20	12.33	4.11	1.680	9.40	.227	.241	Trace.	Trace.		Benzoic acid.	Do.	
19876	Peach ^a	73.54			.211		18.07		+ 97.8 +	73.8	21.5					.316	.271				Benzoic acid.	Do.	
20158	do.....	65.83	.91	.456	.353	28.61	23.65	57.36 +	42.4 +	4.3	22	13.41	4.47		8.57	.430	.341	.062	.010		Benzoic acid.	Do.	
20187	Pear.....	68.34		.175			19.85		+ 80.6 +	54.2	22	50.70	16.90			.385	.281				Salicylic acid. ^a	Do.	
20197	do.....	69.83	1.86	.312	.286	33.66	22.54	56.20 +	39.0 +	8.8	20	19.71	6.57		13.63	.241	.150	.041	.006		Benzoic acid.	Do.	
20021	Plum.....	60.52		.319			1.80		+ 32.4 +	30.0	22	29.43	9.81			.573	.290				Benzoic acid.	Do.	
20028	do. ^a	68.22			.289		8.73		+ 76.5 +	64.9	29.5					.450	.276				Benzoic acid.	Do.	
20148	do. ^a	63.19		.319			None		+ 67.4 +	66.80	20	44.13	14.71			.440	.273				Benzoic acid.	Do.	
20200	do.....	60.35		.456			6.29		+ 30.4 +	22.0	21	16.85	5.95			.392	.279				Benzoic acid.	Do.	
F 652	do.....	73.22				37.10	18.86	55.96 +	68.8 +	44.0	21	(^c)	(^c)		17.26	.336	.225				Benzoic acid.	Do.	
19967	Quince ^a	69.74		.212			1.80		+ 83.0 +	80.6	20	45.18	15.06			.325	.235				Benzoic acid.	Do.	
20202	do.....	65.08	1.33	.206	.333	34.53	15.60	50.13 +	41.5 +	20.6	20	24.42	8.14		14.95	.299	.227	Trace.	.006		Benzoic acid.	Do.	

^a See correspondence with manufacturers, p. 100.

^b The sample was composed in large part of dry, hard berries and consisted entirely of blackberries.

^c Present.

TABLE 24.—Composition of jams containing glucose, but not so labeled—Continued.

Serial number.	Description.	Per cent total solids.	Per cent insoluble solids.	Per cent of protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Approximate per cent glucose (dextrin×3).	Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as per cent potassium sulphate.	Chlorids as per cent sodium chloride.	Starch.	Preserva- tives.	Foreign coloring matter.	
						Per cent reduc- ing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, de- gres C.												
F 637	Quince.....	73.60				22.38	39.70	62.08	+ 72.6	+ 20.4	21	(*)				11.54	0.252	0.095		Small amount.	Benzoic acid.	Red coal tar.	
19963	Raspberry	70.48	1.18	0.281	0.500	36.74	3.28	30.02	+ 88.8	+ 84.4	20	76.68	25.56			30.46	.676	.329	0.630	0.026	Present.	do.	Do.
19879	do.....	74.21			.328		16.79		+ 82.3	+ 60.0	19.5						.413	.295				Salicylic acid.	Pink coal tar.
19959	do.....	64.13			.412				+ 61.7	+ 61.3	19.5						.340	.075				Benzoic acid.	Red coal tar.
19961	do.....	69.65		.350			9.02		+ 72.4	+ 60.4	22	57.00	19.00				.474	.266				Benzoic acid.	Red coal tar.
19966	do. ^b	66.80		.319			5.26		+ 47.2	+ 40.4	22	17.85	5.95				.331	.229				Benzoic acid.	Ponceau red.
20190	do.....	74.04	2.62	.419	.490		4.03	47.29	+ 72.6	+ 67.2	20	49.26	16.42			26.45	.511	.321	.207	.010		Benzoic acid. ^b	Pink coal tar.
20203	do.....	66.18		.525					+ 45.4	+ 25.0	21	23.64	7.88				.360	.284				Salicylic acid. ^b	Pink coal tar.
20585	do.....	72.72	3.74	.700	.588	36.86	15.28	61.24	+ 33.9	+ 1.15	20	11.82	3.94	1.126	11.48		.332	.276	Trace.	.007		do.	Do.
19958	do.....	66.02			.477		24.38		+ 62.0	+ 61.7	20						.510	.118				Benzoic acid.	Red coal tar.
F 654	do.....	74.38				29.77	27.22	56.99	+ 70.4	+ 34.6	21	(*)	(*)		17.39		.372	.260		Consid- erable.	Benzoic acid.	Red coal tar.	
19962	Strawberry	71.32	.66	.319	.510	33.78	9.48	43.26	+ 85.5	+ 75.8	20	69.33	23.11		28.06	.699	.425	.352	.025	Present.	do.	Pink coal tar.	
19964	do.....	70.01			.367		.83		+ 62.9	+ 61.8	19.5						.481	.174				Do.	Do.
19988	do.....	70.00	2.35	.631		37.02	20.60	57.62	+ 31.0	+ 3.4	20	12.33	4.11		12.38	.428	.312	.084	.008			Salicylic acid.	Red coal tar.
20146	do. ^b	66.05		.244	.465		1.05		+ 84.2	+ 82.8	22	57.78	19.26				.413	.293					

20165	do.....	68.83	1.52	.350	.617	58.74	3.28	62.02 +	6.0	—	3.8	20	8.40	2.80		6.81	.385	.241		.003		Orange coal tar.
20167	do.....	70.87	1.77	.594	.534	31.52	29.03	60.55 +	42.9	+	4.0	20	15.72	5.24	2.62	10.32	.476	.341	.111	.006		Salicylic acid.
20205	do.....	65.65		.456			13.33		+ 35.2	+	20.4	21	19.95	6.65			.342	.279				do. ^b ..
21021	do.....	75.56	1.34	.594	.387	38.30	29.77	68.07 +	38.0	—	1.6	20	12.60	4.20		7.49	.388	.308	Trace.	Trace.		Red coal tar.
F 653	do.....	74.18				34.88	17.80	52.68 +	89.8	+	66.4	21	(^a)	(^c)		21.50	.392	.140		None		Benzoic acid.

^a Present.^b See correspondence with manufacturers, p. 101.

Compound jams.—A discussion of the compound jams will serve also for the compound jellies. There is little difference in the formulas given by the manufacturers for the compound jellies and jams, and the difference in their composition is also slight. As a rule, the difference amounts to the addition of a little fruit pulp to the jams. Evaporated apple juice is the basis, to which is added a little of the fruit pulp of the kind desired. The pulp from which the juice has been pressed for making jelly, mixed with glucose and apple juice, makes up many of the compound jams. The consistency of these products is that of thick sirup. No. 19867 has 86.65 per cent of solids and is practically all glucose. The percentage of sugar is practically the same in all compound goods, whether jellies or jams, and the same is true of ash and solids. The nitrogen is a little higher in jams on account of the presence of a small amount of pulp.

TABLE 25.—*Description of compound jams.*

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Blackberry.</i>		
19867	West Virginia Preserving Co., Wheeling, W. Va.	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	Fort Henry Preserves: Fresh fruit, 30 per cent; evaporated apple juice, 25 per cent; corn sirup, 35 per cent; sugar, 10 per cent.
20153	Anderson Preserving Co., Camden, N. J.	Donahue & Kiernan, New York.	Eagle brand compound.
19868	J. Weller Co., Cincinnati, Ohio.	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	60 per cent fruit, 15 per cent sugar, 25 per cent corn sirup; Queen City preserves.
	<i>Currant.</i>		
19874	Vannill Preserving Co., Baltimore, Md.	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	Maryland brand compound.
	<i>Peach.</i>		
20149	Williams Bros. & Charbonneau, Detroit, Mich. ^a	Henry Barge, New York.....	Maple Leaf peach preserves; 50 per cent fruit, 25 per cent glucose, 25 per cent cane.
	<i>Plum.</i>		
19869	West Virginia Preserving Co., Wheeling, W. Va.	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	Fort Henry Preserves: Extra quality fresh fruit, 30 per cent; evaporated apple juice, 25 per cent; corn sirup, 35 per cent; sugar, 10 per cent.
20151	Williams Bros. & Charbonneau, Detroit, Mich. ^a	Henry Barge, New York.....	Maple Leaf brand; 50 per cent fruit, 25 per cent granulated sugar, 25 per cent corn sirup.
20225	Crescent Preserving Co., Camden, N. J.	J. C. Ergood Co., Washington, D. C.	Ivanhoe brand compound.
	<i>Raspberry.</i>		
19969	American Preserve Co.....	H. Punchard & Son, New York.	American compound.

^a See correspondence with manufacturers, p. 104.

TABLE 25.—*Description of compound jams*—Continued.

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturer.
	<i>Strawberry.</i>		
19968	American Preserve Co	H. Punchard & Son, New York.	American compound strawberry jam.
20150	Williams Bros. & Charbonneau, Detroit, Mich.*	Henry Barge, New York.....	Maple Leaf brand; 50 per cent fruit, 25 per cent granulated sugar, 25 per cent corn sirup.
20219	P. J. Ritter Conserve Co., Philadelphia, Pa.	J. C. Ergood Co., Washington, D. C.	Favorite brand pure fruit preserves.
20227	The E. G. Dailey Co., Detroit, Mich.	do	Fresh fruit jam compound; 50 per cent fresh fruit, 15 per cent corn sirup, 35 per cent granulated sugar.

*See correspondence with manufacturers, p. 104.

TABLE 26.—Composition of compound jams.

Serial number.	Description.	Per cent total solids.	Per cent insoluble solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarizations.			Approximate per cent glucose (dextrin×3).	Per cent dextrin.	Per cent total ash.	Alkalinity of ash as per cent, K ₂ CO ₃ .	Sulphates as potassium sulphate.	Chlorids as sodium chlorid.	Starch.	Preservatives.	Foreign coloring matter.
						Per cent cane sugar.	Per cent total sugars.	Per cent reducing sugar.	Direct.	Invert.	Temperature, degrees C.									
19867	Blackberry	86.65		0.350			4.18		+101.6	+96.0	20	91.92	30.64	0.638	0.351	0.129	0.022		Benzoic acid.....	Red coal tar.
20153	do.	74.80	2.16	.419	0.328	30.45	8.80	39.25	+119.8	+108.0	20	71.76	23.92	.582	.366					Do.
19868	do.	62.73	1.92	.387	.588	32.35	1.04	33.39	+86.4	+85.0	20	55.73	18.91	.587	.308	.186	.032		Salicylic acid ^a	Do.
19874	Current	71.50	.70	.350	.402	29.92	None.	29.92	+136.6	+136.2	20	89.55	29.85	.687	.301	.396	.053		do.....	Do.
20149	Peach	66.17		.319			5.89		+96.6	+88.8	20	29.67	9.89	.469	.281	.125	.009			Do.
19869	Plum	74.27		.281					+111.2	+110.0	22	98.76	9.81	.524	.376				Benzoic acid.....	Pink coal tar.
20151	do.	63.41		.244			None.		+83.6	+83.2	20	43.35	14.45	.482	.293					Do.
20225	do.	70.08	.35	.281	.490	39.92	5.52	39.44	+99.2	+91.80	21	84.03	28.01	.502	.357	.084	.054		Benzoic acid.....	Ponceau red.
19969	Raspberry ^a	67.74		.594			None.		+105.0	+104.6	21	61.20	20.40	.657	.391			Present.....	do.....	Do.
19968	Strawberry ^a	71.96		.350			1.19		+111.0	+109.4	20	64.35	21.45	.622	.398			do.....	do.....	Do.
20150	do.	65.94		.244			1.50		+91.0	+89.0	22	46.74	19.26	.461	.263					Red coal tar.
20219	do. ^b	56.41	2.19	.456	.578	33.41	1.17	34.58	+56.40	+54.8	20	37.02	12.34	.582	.357	.085	.006	Present.....	Benzoic acid.....	Do.
20227	do.	73.32		.175			2.99		+116.8	+112.8	20	58.05	19.35	.478	.335				do.....	Do.

^a See correspondence with manufacturers, p. 99.^b See correspondence with manufacturers, p. 103.

JELLIES.

Fifty-eight samples of jellies were analyzed, of which number 44 were commercial products; the remaining 14 were prepared in the laboratory. Of the commercial samples, 19 contained no glucose, 13 contained glucose, but were not so labeled, and the remaining 12 were purchased on the market as compound goods. The general discussion under jams (p. 53 and following) applies also to jellies, owing to the similarity of the products. The jellies are classified upon the same basis as the jams into "Jellies containing no glucose," "Jellies containing glucose, but not so labeled," and "Compound jellies."

Jellies containing no glucose.—Thirty-three of the total number of 58 jellies examined were found to contain no glucose. The amount of solids is extremely variable, the maximum being 80.28 and the minimum 45.56 per cent. The commercial jellies are very much less variable than the homemade products and probably for the reason brought out in Table 31, which shows that the manufacturer, by the use of apple as a basis, is able to produce very uniform products with fruits of widely different gelatinizing powers. The percentage of ash is as variable as the solids, but the alkalinity, sulphates, and chlorids have the same relation to the total ash as in the pure fruit jams.

TABLE 27.—Description of jellies containing no glucose.

Serial number.	Manufacturer.	From whom purchased.	Price.	Claims of manufacturer.
	<i>Apple.</i>			
20023	R. H. Macy & Co., New York....	R. H. Macy & Co., New York.	\$0.29	Macy's crab - apple jelly.
20226	Campbell Preserving Co	J. C. Ergood Co., Washington, D. C.	.10	Apple jelly, lemon flavor.
20408	Made in laboratory			
21062	Dodson, Braun & Co., St. Louis ..	J. G. Swarbrick, 21 Camp street, New Orleans, La.	.20	Apple jelly.
	<i>Blackberry.</i>			
20405	Made in laboratory			
	<i>Crab apple.</i>			
20410	Made in laboratory			
	<i>Currant.</i>			
19890	Hazel Pure Food Co., New York and Chicago.	Siegel, Cooper & Co., New York.	.25	Hazel's currant jelly.
20024	R. H. Macy & Co., New York	R. H. Macy & Co., New York.	.29	Currant jelly.
20169	Miss Martin, Willowbank, N. Y..	C. C. Bryan, Washington, D. C.	.40	Do.
20170	Mrs. S. C. Stone, New Haven, Conn.	do	.40	Spiced currant jelly.
20180	Oneida Community, Ltd., Kenwood, N. Y.	N. W. Burchell, Washington, D. C.	.25	Black currant jelly.
20210	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Red currant jelly.
20214	do	do	.25	Black currant jelly.

TABLE 27.—Description of jellies containing no glucose—Continued.

Serial number.	Manufacturer.	From whom purchased.	Price.	Claims of manufacturer.
	<i>Grape.</i>			
20184	J. P. Smith, New York.....	N. W. Burchell, Washington, D. C.	\$0.25	
20189	Geo. E. Wales, Newton Center Mass.	do.....	.40	
20405	Made in laboratory			
	<i>Guava.</i>			
20246	Jalma Lapham, Indianola, Fla..	Park & Tilford, New York..	.35	
21084	S. W. Clark & Son.....	A. M. & J. Solari, Royal and Customhouse streets, New Orleans, La.	.40	
	<i>Huckleberry.</i>			
20412	Made in laboratory			
	<i>Lemon.</i>			
20173	Gordon & Dilworth, New York..	C. C. Bryan, Washington, D. C.	.30	
	<i>Orange.</i>			
20435	Made in laboratory			
	<i>Peach.</i>			
20437	Made in laboratory			
	<i>Pear.</i>			
20434	Made in laboratory			
	<i>Pineapple.</i>			
20436	Made in laboratory			
	<i>Pineapple husk.</i>			
20433	Made in laboratory			
	<i>Plum.</i>			
20404	Made in laboratory			
20409	do.....			
20411	do.....			
	<i>Raspberry.</i>			
20185	J. P. Smith, New York	N. W. Burchell, Washington, D. C.	.25	Raspberry.
20188	Geo. E. Wales, Newton Center, Mass.	do.....	.40	Raspberry jelly.
	<i>Strawberry.</i>			
20172	Gordon & Dilworth, New York ..	C. C. Bryan, Washington, D. C.	.30	Strawberry.
20186	J. P. Smith, New York.....	N. W. Burchell, Washington, D. C.	.25	Do.
	<i>Mixed fruit.</i>			
20407	Made in laboratory			

TABLE 28.—Composition of jellies containing no glucose

Serial number.	Description.	Per cent total solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as potassium sulphate.	Chlorids as sodium chlorid.	Starch.	Preservatives.
					Per cent reduced sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, degrees C.									
20023	Apple	62.67	0.175	0.255	34.08	25.45	59.53	+16.20	-17.90	20		0.51	3.11	0.100	0.150	Trace.	Trace.		Salicylic acid.
20226	do	66.06	.112	.608	51.52	11.53	63.05	- 6.90	-22.35	20		1.13	2.90	.260	.248	Trace.	0.011		
20408	do	59.18	.175	.279	20.78	33.04	53.82	+24.00	-20.60	18			5.36	.220					
21062	do	60.86	.213	.686	37.50	18.13	55.63	+ 4.00	-20.30	20		1.30	5.23	.354	.302	0.054	Trace.	Present.	
20405	Blackberry	59.63	.243	.475	12.51	44.90	57.41	+47.00	-20.10	18			2.22	.330					
20410	Crab apple	63.28	.137	.171	34.93	23.68	58.61	+13.00	-19.00	18			4.67	.110					
19890	Currant	64.21	.312	1.362	54.37	4.70	59.00	-11.80	-18.10	20		3.36	5.14	.438	.416	.035	Trace.		
20024	do	62.46	.175	1.058	52.74	7.16	59.90	- 9.10	-18.70	20		1.39	2.52	.272	.264	Trace.			Do.
20169	do	66.22	.212	.923	54.20	11.50	65.70	- 5.90	-21.30	20		1.18	.52	.222	.221	Trace.	Trace.		
20170	do	70.31	.281	.926	58.79	7.09	65.79	-10.50	-20.00	20		2.11	4.43	.384	.312	Trace.	Trace.		Do. ^a
20180	do	75.03	.212	1.146	46.56	22.69	69.25	+ 8.10	-22.30	20		2.87	5.78	.474	.436	.054	Trace.		Benzoic acid.
20210	do	68.77	.212	1.078	53.97	10.52	64.49	- 7.90	-22.00	20		1.70	4.28	.414	.383	Trace.	Trace.		
20214	do	59.37	.312	1.573	51.20	3.96	55.16	-12.90	-18.20	20			4.21	.624	.511	.058	Trace.		
20184	Grape	70.66	.175	.314	49.76	14.37	64.13	+ .15	-19.15	20		1.25	6.53	.214	.276	Trace.	Trace.		
20189	do	72.22	.212	.833	64.27	3.47	67.74	-17.25	-21.90	20		2.49	4.48	.346	.234	Trace.	Trace.		
20405	do	63.66	.175	.524	32.29	30.52	62.81	+22.30	-18.90	18			.85	.450					
20246	Guava	79.97	.175	.470	31.46	48.43	74.89	+33.40	-24.80	20		1.36	5.08	.448	.414	Trace.	Trace.		
21084	do	79.14	.175	.470	30.66	44.25	75.91	+35.20	-24.10	20		1.59	4.23	.384	.349	Trace.	Trace.		
20412	Huckleberry	63.02	.069	.245	24.27	32.74	57.01	+24.10	-20.10	18			.280						
20173	Lemon	64.62	.112	.456	54.18	7.31	61.49	-10.00	-19.80	20		.73	3.13	.206	.194	Trace.	Trace.		
20435	Orange	68.56	.418	.171	3.95	62.52	66.47	+61.30	-23.10	18			2.09	.300					
20437	Peach	69.98	.175	.245	8.75	56.59	65.34	+53.40	-23.00	18			4.64	.210					
20434	Pear	69.12	.156	.181	6.58	58.46	65.04	+52.70	-26.20	18			4.08	.340					
20436	Pineapple	80.28	.387	.328	22.13	56.70	78.83	+50.40	-26.10	18			1.45	.430					

^aSee correspondence with manufacturers, p. 103.

TABLE 28.—Composition of jellies containing no glucose—Continued.

Serial number.	Description.	Per cent total solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.			Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as potas- sium sulphate.	Chlorids as sodium chlorid.	Starch.	Preservatives.
					Per cent reduc- ing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, degrees C.								
20433	Pineapple husk . . .	76.34	0.350	0.352	7.40	65.22	72.62	+63.70	-24.30	18		3.72	0.730					
20404	Plum	45.56	.350	1.127	19.18	22.67	41.85	+17.80	-12.80	18		3.71	.680					
20409	do	54.49	.138	1.029	24.00	25.48	49.48	+16.70	-17.80	18		5.01	.400					
20411	do	73.01	.175	1.529	44.22	22.37	66.59	+ 7.60	-22.60	18		6.42	.650					
20185	Raspberry	70.07	.175	.333	46.46	16.79	63.25	+13.70	- 8.80	20		1.45	.244	0.257	Trace.	Trace.		
20188	do	74.53	.312	.828	65.52	5.07	70.59	-14.00	-20.80	20		.77	.310	.241	Trace.	Trace.		
20172	Strawberry	68.11	.175	.289	40.83	22.24	63.07	+12.20	-17.60	20		.89	.208	.207	Trace.	Trace.		
20186	do	67.50	.175	.294	44.56	18.00	63.56	+ 7.50	-16.60	20		1.47	.210	.311	Trace.	Trace.		
20407	Mixed fruit	66.58	.069	.367	39.70	24.22	63.92	+14.80	-17.90	18		2.66	.210					
	Average	67.13	.208	.633	37.07	25.96					2.53	4.05	.345	.302	.011	.001		
	Maximum	80.28	.418	1.573	65.52	65.22					3.36	6.82	.730	.511	.058	.011		
	Minimum	45.56	.069	.171	3.95	3.47					.73	.52	.100	.150	Trace.	Trace.		

The polarization after fermentation in all these jellies was practically zero, showing that there was no dextrin present. The percentage of sugar-free solids is fairly uniform, averaging 4.05 per cent, with a maximum of 6.82. The value of this fact will be seen in dealing with the commercial jellies containing glucose in which the sugar-free solids may be from 30 to 40 per cent.

The protein content is chiefly of value in the detection of the addition of gelatin, but in all the samples examined, of all grades and kinds, there were none that gave any indication of having received additions of gelatin.

The average content of protein is but 0.208 per cent, and it would take only a very slight addition of gelatin to raise the protein content much above the normal. It is very probable that gelatin would not be used for other reasons, since it spoils very easily.

The most striking fact brought out by the table is that none of these jellies shows any added coloring matter. They are the highest grade products, except that some of them contain preservatives, and this would indicate that a good product can be made on a commercial scale without the use of glucose or coloring matter. This also disproves the claim made by some manufacturers that it is necessary to add glucose to jellies in order to prevent crystallization of the sugar. In the jellies in this table the sugar content ran as high as 74 per cent, and in only two cases was there any crystallization of sugar. These were 20188, having 70.59 per cent of total sugar, of which 65.52 per cent was reducing sugar, and 20170, with 65.88 per cent of total sugar, of which 58.79 per cent was reducing sugar. In both cases it was the dextrose that had crystallized.

To show the uniformity of jellies from one manufacturer, and also to call attention to the desirability of comparing closely the products of a single manufacturer, the following table has been prepared, which shows that it is possible for the manufacturer to obtain from strawberries and raspberries, two fruits which jelly only with difficulty, as firm a jelly as can be made from the apple and the quince, which are considered the best jelling fruits.

TABLE 29.—*Analyses of the products from one manufacturer.*

Samples.	Solids.	Ash.	Protein N $\times$ 6.25.	Acid, as H ₂ SO ₄ .	Total sugar.	Dextrin.	Alcohol precipitate.	Sugar-free solids.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Apple	64.46	0.210	0.249	0.625	57.00		4.22	7.46
Grape	63.26	.342	.216	.634	54.76	2.94	4.35	8.50
Quince	60.48	.355	.139	.510	52.34	3.22	3.31	8.14
Raspberry	61.28	.316	.178	.574	55.04	3.31	4.01	6.24
Strawberry	62.94	.387	.249	.684	52.60	5.10	3.46	10.34

All these jellies were uniform in consistency and appearance, except in color, and the analyses show practically the same percentage of solids and sugar. When we consider that the apple and the strawberry are nearly opposites as far as gelatinizing power goes, it is remarkable that this can be accomplished with the same amount of sugar. The explanation is simple. There is little doubt that apple is the basis of all these products, and only enough of the fruit indicated on the label is added to give the desired flavor.

Jellies containing glucose but not so labeled.—There is little to be said about the jellies included in these tables. They embrace jellies of nearly all grades, from the cheapest to the highest priced. Six contain only very small amounts of glucose, which may have been added for the purpose of preventing the crystallization of the cane sugar. But the firm putting out these goods have since decided that glucose is not necessary for this purpose and claim to have given up its use. What has been said in the general discussion of jams and jellies applies here as well. Five of these jellies were colored and nine contained preservatives.

TABLE 30.—*Description of jellies containing glucose but not so labeled.*

Serial number.	Manufacturer.	From whom purchased.	Price.	Claims of manufacturers.	Remarks.
	<i>Apple.</i>				
20174	-----	C. C. Bryan, Washington, D. C.	\$0.25	Apple.....	Full of coarse particles; moldy on top.
20209	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Pure apple jelly.	
	<i>Currant.</i>				
20221	P. J. Ritter Conserve Co., Philadelphia, Pa.	J. C. Ergood & Co., Washington, D. C.	.15		
	<i>Grape.</i>				
20213	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Pure grape jelly.	
F 662	Sprague, Warner & Co., Chicago, Ill.	See & Co., Chicago, Ill.	.10	Grape; Manhattan brand fresh fruit jelly. Packed from choicest stock.	
	<i>Guava.</i>				
20996	Viaberay & Co., Habana.	A. M. & J. Solari, New Orleans, La.	.20		
	<i>Lemon.</i>				
19882	Hazel Pure Food Co., New York and Chicago.	Siegel - Cooper Co., New York.	.25	Hazel lemon jelly ..	Strongly flavored with lemon.
F 661	Sprague, Warner & Co., Chicago, Ill.	See & Co., Chicago, Ill.	.10	Lemon jelly; Manhattan brand fresh fruit jelly. Packed from choicest fruit.	

TABLE 30.—*Description of jellies containing glucose, but not so labeled*—Continued.

Serial number.	Manufacturer.	From whom purchased.	Price.	Claims of manufacturers.	Remarks.
<i>Plum.</i>					
19887	Milford Manufacturing Co., Chicago.	Siegel - Cooper Co., New York.	\$0.17		Moldy on top; also contained large number of crystals of acid tartrate of potash.
<i>Quince.</i>					
20212	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Pure quince jelly.	
20222	P. J. Ritter Conserve Co., Philadelphia, Pa.	J. C. Ergood & Co., Washington, D. C.	.15		
F 660	Sprague, Warner & Co., Chicago, Ill.	See & Co., Chicago, Ill.	.10	Quince jelly; Manhattan brand fresh fruit jelly. Packed from choicest fruit.	
<i>Raspberry.</i>					
19886	Milford Manufacturing Co., Chicago.	Siegel - Cooper Co., New York.	.17	Raspberry jelly.	
20154	Ayer Preserving Co., Ayer, Mass.	Donahue & Kiernan, New York.	.12		
20211	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Pure raspberry jelly	
F 658	Sprague, Warner & Co., Chicago, Ill.	See & Co., Chicago, Ill.	.10	Raspberry jelly; Manhattan brand fresh fruit jelly. Packed from choicest fruit.	
<i>Strawberry.</i>					
20208	Curtice Bros. Co., Rochester, N. Y.	G. G. Cornwell & Son, Washington, D. C.	.25	Pure strawberry jelly.	

TABLE 31.—Composition of jellies containing glucose but not so labeled.

Serial number.	Description.	Per cent total solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarizations.		Approximate per cent glucose (dextrin×3).	Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Sulphates as per cent potassium sulphate.	Chlorides as per cent sodium chloride.	Starch.	Preserva- tives.	Foreign color- ing matter.
					Per cent cane sugar.	Per cent total sugars.	Direct	Invert.	Tempera- ture,°											
20174	Apple.....	65.57	0.281	0.614	38.13	None.	114.7	+114.5	20	71.7	23.90	9.61	27.44	0.392	0.276	0.110	0.011		Saccharin.	
20209	do.....	63.19	.244	.613	38.13	17.76	35.0	+11.2	20	Present. ^a		4.14	7.30	.206		.076	Trace.	Present.	Benzoic acid.	
20221	Curant ^b	71.86	.175	.417	37.66	17.24	58.9	+35.8	20	31.50	10.50	11.37	16.96	.444	.376	.088	.011	Present.	do.....	Coal-tar dye.
20213	Grape.....	62.02	.212	.622	44.35	9.37	7.2	-5.35	20	8.67	2.89	4.27	8.30	.336	.330	Trace.	Trace.	Present.	Salicylic acid. ^a	Cochineal.
F662 ^c	Grape.....	61.87			33.93	1.50	35.43	+93.8	21				26.44	.686	.071			Small amt.	Benzoic acid.	Red coal tar.
20996	Guava.....	78.57	.175	.450	36.32	32.09	39.0	-4.0	20	11.82	3.94	9.69	10.36	.538	.422	.112	.010			
19882	Lemon.....	67.74	.281	.607	53.76	4.33	58.09	+22.0	20	19.95	6.65	8.11	9.65	.262	.253	.026	.020			
F661 ^d	do.....	61.03			30.16	None.	103.8	+102.2	21				30.87	.419	.225			(^e)	Benzoic acid.	Yellow coal tar.
19887	Plum.....	66.58	.175	.897	37.02	None.	97.2	+97.4	20	72.75	24.25	31.50	29.96	.387	.315	.059	.034	Trace.	Benzoic acid.	Do.
20212	Quince.....	59.27	.137	.500	34.91	16.42	17.0	-5.0	20	9.48	3.16	3.25	7.94	.348	.315	Trace.	.005		Benzoic acid.	
20222	do. ^b	65.92	.175	.402	34.56	17.01	53.8	+31.0	20	27.06	9.02	11.77	14.35	.422	.319	.088	.010	Present.	do.....	Slight coal tar.
F660 ^f	Quince.....	60.82			32.45	None.	100.4	+100.4	21				28.37	.715	.002			Abun- dant.	do.....	
19886	Raspberry.....	62.80	.212	.662	38.53	None.	68.2	+68.2	20	52.53	17.51	14.98	24.29	.500	.290	Trace.	Trace.	Present.		
20154	do.....	72.67	.244	.607	46.13	1.20	84.20	+82.6	20		13.31	14.26	25.34	.386	.338	Trace.	.052	Present.	Salicylic acid.	
20211	do.....	60.08	.175	.563	44.26	9.70	9.0	-4.0	20	9.75	3.25	3.93	6.12	.310	.259	Trace.	.005	Present.	Benzoic acid.	Do.
F658 ^g	Raspberry.....	62.97			32.53	1.05	102.6	+101.2	21				29.39	.684	.106			Abun- dant.	do.....	Red coal tar.
20208	Strawberry.....	61.70	.244	.671	47.84	3.73	10.6	+5.6	20	15.00	5.00	3.40	10.13	.380	.338	Trace.	.017	Present.	Salicylic acid. ^a	Do.

^a See correspondence with manufacturers, page 101.^b See correspondence with manufacturers, page 103.^c Sample contains 0.17 per cent phosphoric acid.^d Sample contains 0.04 per cent phosphoric acid.^e Very strong test.^f Sample contains 0.14 per cent phosphoric acid.^g Sample contains 0.15 per cent phosphoric acid.

Compound jellies.—The table of compound jellies includes only the goods marked compound. The formulæ for eight of these samples are given, and according to these there is great similarity in composition. All claim at least 50 per cent of fruit juice, mostly apple, from 5 to 10 per cent of cane sugar, and from 35 to 45 per cent of glucose. A jelly prepared according to such a formula would contain less than 50 per cent of solids, whereas the majority of the samples examined contained over 70 per cent. It is therefore apparent from the percentage of solids that, if the formula is correctly represented on the label, the products have either been highly concentrated by evaporation or else the apple juice was very concentrated.

Only two samples, 20223 and 20224, were actually jellies; the others were thick sirups with not the slightest appearance of jelly. If these jellies were boiled down after the sugar was added it might account for the entire lack of any cane sugar in the final product; but if highly concentrated apple juice was used and only heated a little, as is ordinarily the case in making jellies, there should be some of the cane sugar uninverted, especially where, as in 20232, it is claimed that sugar has been added. The percentage of dextrin in some of these products is very high, and it is evident that the factor 3 can not be used for the calculation of the approximate per cent of glucose. It is probable that in the preparation of such samples confectioners' glucose has been used, which is not completely converted, and consequently the product has a very high dextrin content. But even using the factor 2 to determine the amount of added glucose, a factor which is very much lower than that given by any glucose product described by Saare,^a the amount of glucose is still much higher than is indicated in the formula. The ash also indicates that a very different kind of glucose has been used from that employed in the samples mentioned in Table 4.

It is said that some manufacturers of cheap jellies use starch as a base, which they partially hydrolyze with phosphoric or sulphuric acid, without purifying the product in any way, except neutralizing the acid.

In seven of these samples the amount of sulphates in the ash is very much higher as a rule than in the pure glucose ashes in the above-mentioned table. As might be expected, starch was found in nearly all the compound jellies. The source of the starch was probably the apple used as the basis. In case confectioners' glucose is used, however, it may give an appreciable starch reaction.

^a O. Saare, *Die Ind. der Stärke und Stärkefabrikate in den Vereinigten Staaten von Amerika*. Berlin, 1896.

TABLE 32.—*Description of compound jellies.*

Serial number.	Description and manufacturer.	From whom purchased.	Price.	Claims of manufacturer.	Remarks.
19865	<i>Apple.</i>				
20224	P. J. Ritter Conserve Co., Philadelphia.	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	\$0.20	50 per cent evaporated apple juice, 45 per cent glucose, 5 per cent sugar.	Large amount of insoluble matter present; 3-pound pail, 20 cents.
20232	The E. G. Dailey Co., Detroit.	J. C. Ergood & Co., Washington, D. C.	.10	Strawberry flavor; Favorite brand compound.	
19864	<i>Currant.</i>				
19970	Grocers Preserve Co., Boston	Park & Tilford, New York		Compound, 50 per cent evaporated apple juice, 40 per cent corn sirup, 10 per cent sugar.	
20155	E. G. Dailey Co., Detroit	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.	.15	50 per cent evaporated apple juice, 45 per cent glucose, 5 per cent sugar.	Large amount of insoluble matter present; 3-pound pail, 15 cents.
20156	Ayer Preserve Co., Ayer, Mass.	H. PUNCHARD & SON, New York	.18	do.....	Large amount of insoluble matter present; 2 pounds for 18 cents. Moldy on top.
20216	American Preserve Co., Philadelphia.	Donahue & Kiernan, New York	.12	40 per cent evaporated apple juice, 10 per cent fruit, 35 per cent glucose, 15 per cent sugar.	
20223	P. J. Ritter Conserve Co., Philadelphia.	do.....	.15	50 per cent evaporated apple juice, 45 per cent glucose, 5 per cent sugar.	Large amount of insoluble matter present; 3-pound pail, 15 cents.
19971	<i>Grape.</i>	J. C. Ergood & Co., Washington, D. C.	.10	Compound jelly.....	
20217	<i>Peach.</i>	do.....	.10	Currant flavor; Favorite brand compound.	
20157	American Preserve Co., Philadelphia, Pa.	H. PUNCHARD & SON, New York	.18	Flavored grape jelly; 50 per cent evaporated apple juice, 45 per cent glucose, 5 per cent sugar.	2 pounds for 18 cents
	<i>Strawberry.</i>	J. C. Ergood & Co., Washington, D. C.	.10	Compound jelly.....	
		Donahue & Kiernan, New York	.15	50 per cent evaporated apple juice, 45 per cent glucose, 5 per cent sugar.	Large amount of insoluble matter present; 3-pound pail, 15 cents.

TABLE 33.—Composition of compound jellies.

Serial number.	Description.	Per cent total solids.	Per cent of protein. (N × 6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarization.		Per cent glucose (dextrin × 5).	Per cent dextrin.	Per cent alcohol precipitate.	Per cent sugar-free solids.	Per cent total ash.	Alkalinity of ash as per cent K ₂ CO ₃ .	Per cent potassium sulphate.	Per cent sodium chloride.	Starch.	Preserva- tives.	Foreign coloring matter.
					Per cent reduc- ing sugars.	Per cent cane sugar.	Total sugar.	Direct.	Invert.											
19865	Apple	75.05	0.244	0.745	37.44	None.	27.44	+131.80	+131.8	20	35.98	8.22	37.61	0.648	0.146	0.546	0.005	Present...		Red coal tar.
20224	do. b	65.34	.244	.515	35.70	4.44	40.14	+99.85	+94.00	20	21.45	25.20	25.20	.616	.396	.216	Trace.	Present...	Benzoic acid.	Do.
20232	do. c	63.82		.592		None.		+119.20	+119.2	21				.538	.245					Do.
19864	Current	77.02	.244	.686	37.66	None.	37.66	+136.2	+136.2	20	31.34	39.83	39.36	.644	.215	.498	.027	Present...		Do.
19970	do. c	70.80	.244	.588	30.53	None.	30.53	+133.6	+134.4	20	30.88	40.63	40.27	.626	.374	.058	Trace.	Present...		Do.
20155	do	69.65	.244	.598	44.03	1.34	45.37	+78.30	+76.50	20	43.86	14.62	13.20	.576	.307	.200	.029	Present...	Salicylic acid.	Do.
20156	do	76.37	.175	.549	29.73	None.	29.73	+153.8	+152.4	20	38.25	43.84	46.64	.648	.229	.448	.011	Present...		Do.
20216	do. d	74.01	.212	.304	27.44	21.20	48.54	+100.30	+71.90	20		23.43	25.37	.379	.314	Trace.	.090			Do.
20223	do. b	65.53	.350	.418	30.94	4.92	35.86	+96.30	+88.70	20	63.90	21.30	29.69	.736	.400	.344	.060		Benzoic acid.	Do.
19971	Grape	72.77	.212	.578	31.84	None.	31.84	+141.40	+141.40	20	30.81	38.05	40.93	.636	.395	.179	.005	Present...		Do.
20217	Peach ^a	70.04	.244	.318	32.32	14.74	47.06	+85.35	+65.65	20		16.49	22.98	.336	.192	Trace.	.040	Present...		Do.
20157	Strawberry ...	75.90	.175	.686	29.98	None.	29.98	+159.2	+159.2	20	39.14	40.99	45.92	.688	.229	.557	.016	Present...		Do.

^a A grade of glucose was used having very high content of dextrin.^b See correspondence with manufacturers, p. 103.^c See correspondence with manufacturers, p. 102.^d See correspondence with manufacturers, p. 99.

Six of these jellies are important enough to be put in a separate table, as they illustrate several points very well. They are the cheapest jellies on the market, not costing at retail more than 5 cents a pound. Nos. 19964, 19965, 19970, 19971, 20156, and 20157 are products of three different factories. The glucose used in 19970 and 19971 is evidently not the same grade or made by the same process of manufacture as that employed in the others, as its ash is entirely different. In four of these the percentage of dextrin exceeds that of the sugars. Apparently they are glucoses much like the confectioners' glucose, of which 46 to 48 per cent of the solids are sugar and there is still a slight starch reaction.

TABLE 34.—*Six of the cheapest jellies.*

Serial number.	Solids.	Ash.	Sulphates as potassium sulphate.	Chlorids as sodium chlorid.	Total sugar.	Polariza- tion.	Sugar- free solids.	Dextrin.
19864.....	77.02	0.644	0.498	0.027	37.66	136.2	39.36	31.34
19865.....	75.05	.648	.546	.005	37.44	131.8	37.61	35.98
19970.....	70.80	.626	.058	Trace.	30.53	133.6	40.27	30.88
19971.....	72.77	.636	.179	.005	31.84	141.4	40.93	30.81
20156.....	69.65	.576	.448	.011	29.73	153.8	46.64	38.25
20157.....	75.90	.686	.557	.016	29.98	159.2	45.92	39.14

CANNED FRUITS.

Fruits put up in hermetically sealed receptacles of either glass or tin are so distinctly in a class by themselves that the discussion already given of jams and jellies is applicable only so far as it concerns the original fruit and the detection of the various forms of adulteration. In the manufacture of jams and jellies the tendency is to produce a material of uniform consistency, and to do this it is necessary that the products have a somewhat constant composition. With canned fruits, on the other hand, consistency is not an essential feature, and the ingredients added, depending upon the product desired, may vary within wide limits. Goods of this class do not yield themselves so readily to adulteration as do those already considered. They are preserved whole or in pieces of considerable size. The general appearance of the fruit is thus retained, and by means of a macroscopic examination the analyst can detect the presence of foreign fruit. Thus the substitution of cheap fruit for the more expensive kinds is not practiced here.

The most important considerations in the examination of canned fruits are: The general quality of fruit employed, the density of sirup, the presence or absence of such materials as glucose, saccharin, foreign coloring matter and preservatives, and the fidelity of label regarding the variety of fruit employed and the name and location of manufacturer.

The fruits examined are described in Table 35, following.

TABLE 35.—Description of canned fruit.

Serial number.	Description and manufacturer.	From whom purchased.	Claims of manufacturers.	Remarks.
	<i>Apples.</i>			
20236	Dale Bros., Oneida County, N. Y.	Park & Tilford, New York.....	Clover brand apples.....	
	<i>Apricots.</i>			
19900	Vacaville Preserving Co., Sacramento Cal.	Siegel-Cooper Co., New York.....	Royal Red apricots, finest quality.....	
20239	Maryville Fruit Packing Company, Maryville, Cal.	Park & Tilford, New York.....	Extra dessert fruit.....	
20611	August Schmidt.....	R. H. Macy & Co, New York.....	Aprikosen.....	
21049	L. Morel, Posen, Germany.....	J. G. Swarbrick, 321 Camp street, New Orleans, La.	Apricots.....	
	<i>Blackberries.</i>			
19892	Gibbs Preserving Co., Baltimore, Md.....	Siegel-Cooper Co., New York.....	Lawton dessert blackberries.....	
	<i>Blueberries.</i>			
20249	J. & E. A. Wyman, Cherryfield, Me.....	Park & Tilford, New York.....	Extra quality Maine blueberries.....	
	<i>Cherries.</i>			
19885	Hazel Pure Food Co., New York and Chicago.	Siegel-Cooper Co., New York.....	Red cherries.....	
20195	Gordon & Dilworth, New York.....	G. G. Cornwell & Son, Washington, D. C.	Fresh red cherries.....	
20240	Hooven Mercantile Co., New York.....	Park & Tilford, New York.....	Bonita brand.....	
20242	Mack & Lyon, Detroit, Mich.....	do.....	Fresh cherries.....	
20243	Ellen H. North, Genesee, N. Y.....	do.....	Red cherry preserves.....	
20248	Wayne Preserving Co., Fairport, N. Y.....	do.....	Pitted cherries.....	
20546	W. Laaff, Mainz, Germany.....	Hanscom Bros., 1311 Market street, Philadelphia.	Kirschen.....	
19895	Oneida Community, Ltd., Kenwood, N. Y.	Siegel-Cooper Co., New York.....	Choice red pitted cherries.....	
19898	Sacramento Preserving Co., Sacramento, Cal.	do.....	Royal Red brand black cherries (all goods under this brand guaranteed).	
20136	Sutter Canning and Packing Co., Yuba City, Cal.	Henry Barge, New York.....	Extra California black cherries.....	

TABLE 35.—*Description of canned fruit—Continued.*

Serial num-ber.	Description and manufacturer.	From whom purchased.	Claims of manufacturers.	Remarks.
	<i>Cherries—Continued.</i>			
20138	Seeman Bros., New York.....	Henry Barge, New York.....	Waverly California white cherries...	
20230	G. Amandus, Mainz, Germany.....	Park & Tilford, New York.....	Red cherries	
20834			Compote de Cerises au Sirop guaranti pur sucre.	
21035	Augustus Besse, Bordeaux.....	Popovich & Abramovich, New Orleans, La.	Bigarreaux au Marasquin	
21052	La Porte Frère et Fils, Paris.....	J. G. Swarbrick, New Orleans, La	Cerises—Bigarreaux au Marasquin...	
21053	Moray Fils et Cie, Lyons	do.....	Bigarreaux au Marasquin	
21079	G. Amandus, Mainz.....	S. W. Clark & Son, New Orleans, La	Kirschenweiss.....	
21085	Teyssonneau, Jeune, Bordeaux.....	do.....	Bigarreaux au Marasquin.....	
	<i>Figs.</i>			
19897	G. W. Dunbar's Sons, New Orleans, La	Siegel-Cooper Co., New York	Fresh ripe figs.....	
21086	Teyssonneau, Jeune, Bordeaux.....	S. W. Clark & Son, New Orleans, La.....	Figs.....	
	<i>Grapes.</i>			
20231	A. Lusk & Co., California Canning Co., Cal.....	Park & Tilford, New York.....	Bear Brand Muscat grape.....	
	<i>Oranges.</i>			
21088	G. W. Dunbar & Sons, New Orleans, La....	S. W. Clark & Son, New Orleans, La.....	Fresh fruit preserved in heavy sirup ..	
	<i>Peaches.</i>			
20193	Jos. Campbell Co., Camden, N. J	N. W. Burchell, Washington, D. C	Preserved peaches.....	
19866	Jas. Madison, San Francisco, Cal.....	R. W. Crounse, 950 Louisiana ave, Wash- ton, D. C.....	Choice California standard fruit	
19871	Cosmos Packing Co., San Francisco, Cal.....	do.....	Cosmos brand peaches, extra stand- ard quality.	
19893	San Leandro Packing Co., San Leandro, Cal.....	Siegel-Cooper Co., New York	Selected fruit packed in pure white sugar sirup, Holly brand.	
20233	Sacramento Packing Co., Sacramento, Cal.....	Park & Tilford, New York	Derby lemon cling peaches, extra standard California fruits.	

				Cork layer between lead top and fruit.
21046	Dandicolle & Gaudin, Bordeaux..... <i>Pears.</i>	J. G. Swarbrick, New Orleans, La.		
20140	Golden Gate Packing Co., Yuba City, Cal.	Henry Barge, New York		Golden State extra Bartlett pears. Selected California fruit put up in heavy sirup.
20144	Francis H. Leggett & Co., New York	do.		Ceres brand Bartlett pears
20832	Felix Potin.....	E. R. Lake, Corvallis, Oreg		Compote de poires au sirop guaranti pur sucre.
19870	Thos. J. Meyer & Co., Baltimore, Md.....	R. W. Crounse, 950 Louisiana avenue, Washington, D. C.		Maryland brand Bartlett pears; first quality.
19872	—, San Francisco, Cal.....	do.		Iron city brand Bartlett pears, extra standard. Guaranteed.
19899	Oregon Packing Co., Portland, Oreg.....	Siegel-Cooper Co., New York		Oregon brand Bartlett pears from selected graded fruits packed in best granulated sugar sirup.
20134	B. S. Ayar, Bridgeton, N. J	Henry Barge, New York.....		Jersey pears, a superior quality. A brand selected with great care.
20137	Flour City Packing Co., Rochester, N. Y.	do.		Flour city brand Bartlett pears. All goods bearing this brand are guaranteed equal to any so-called standards.
20235	The J. H. Flickinger Co., San Jose, Cal ...	Park & Tilford, New York.....		Bartlett pears. Pure sugar sirup, extra table fruit; put up ripe in orchard where grown.
20009	Henry Doraix	R. H. Macy & Co., Fourteenth street, New York.		Conservierte Früchte (pears), Eugene Duraix.
21048	Dandicolle & Gaudin, Bordeaux.....	J. G. Swarbrick, New Orleans, La.		Do.
19875	H. Masten, jr., Felton, Del	R. W. Crounse, Washington, D. C.		Pears. Pure sugar sirup
19896	Hazel Pure Food Co., New York.....	Siegel-Cooper Co., New York		Grated pineapple, finest quality
20135	F. H. Leggett & Co., New York	Henry Barge, New York		Premier pineapple
20143	R. C. Williams & Co., New York	do.		Sliced pineapple
20234	Gordon & Dilworth, New York	Park & Tilford, New York		First quality pineapple.....

TABLE 35.—*Description of canned fruit*—Continued.

Serial number.	Description and manufacturer.	From whom purchased.	Claims of manufacturers.	Remarks.
<i>Pineapple</i> —Continued.				
20228	E. G. Dailey Co., Detroit, Mich.	Park & Tilford, New York.	Poyet pineapple preserves.	
20612	Eugene Duralix, Hoflieferant.	R. H. Macy & Co., New York.	Pineapple Conservierte Früchte.	
<i>Plums.</i>				
19894	Millford Manufacturing Co., Chicago, Ill.	Siegel-Cooper Co., New York.	Extra standard, Millford egg plums.	
20139	Seeman Bros., New York.	Henry Barge, New York.	Green gage plums, Waverley brand.	
21250	Moral's Conserven, Berlin.	Park & Tilford, New York.	Geschälte Pfäumen.	
20142	Golden Gate Packing Co., Yuba City, Cal.	Henry Barge, New York.	Green gage plums, Golden State extra selected California fruits packed in heavy sirups.	
20238	Golden Gate Packing Co., San Jose, Cal.	Park & Tilford, New York.	Green gage plums. Selected California fruit. Packed for Park & Tilford.	
20836	Felix Potin.	E. R. Lake, Exp. Sta., Corvallis, Ore.	Reines Claude.	
21047	Dandicolle & Gaudin, Bordeaux.	J. G. Swarbrick, Camp street, New Orleans, La.	Prunes.	Cork layer between lead top and fruit.
<i>Raspberries.</i>				
21051	S. Morel, Posen, Germany.	do.	Framboises au jus.	
21078	Gustav Amandus, Hoflieferant, Wiesbaden.	S. W. Clark & Son, 624 Canal street, New Orleans, La.	Himbeeren.	
20191	L. Bradbury & Son, Webbs Mills, N. Y.	N. W. Burchell, Washington, D. C.	Willow Bank brand raspberries.	
<i>Strawberries.</i>				
20229	G. Amandus, Mainz, Germany.	Park & Tilford, New York.	Ananas erdbeeren.	
20344	W. Laaff.	Hanscom Bros., Philadelphia, Pa.	Ananas erdbeeren.	
20610	August Schmidt, Hoflieferant, Wiesbaden.	R. H. Macy & Co., New York.	Erdbeeren.	
20953	Carl Berg.	A. M. & J. Solari, New Orleans, La.	Erdbeeren.	
21045	Teyssonneau, Jeune, Bordeaux.	J. G. Swarbrick, New Orleans, La.		
21050	S. Morel, Posen, Germany.	do.	Cerise.	

Miscellaneous.

19891	Cape Cod Preserving Co.....	Siegel-Cooper Co., New York	Choice Cape Cod cranberry sauce.....
20247	Bishop & Co., Los Angeles, Cal	Park & Tilford, New York	Pickled figs.....
20164	do	C. C. Bryan, Washington, D. C	Pickled watermelon.....
20545	W. Laaff, Mainz.....	Hanscom Bros., Philadelphia, Pa	Melange.....
20244		Park & Tilford, New York	Peach chutney.

TABLE 36.—Composition of canned fruits.

Serial number.	Description of sample.	Total solids.		Per cent insoluble solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarizations.			Per cent dextrin.	Approximate per cent glucose (dextrin×3).	Per cent total ash.	Alkalinity as per cent K ₂ CO ₃ .	Sulphates as per cent potassium sulphate.	Chlorids as per cent sodium chlorid.	Preservatives.	Foreign coloring matter.
		Per cent in entire contents of can.	Per cent in slup.				Per cent reduced sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, degrees C.								
20236	Apples	8.17						None.		4.4	4.5	19			0.147	0.115				
19900	Apricots	16.84				0.882		0.59		3.2	4.0	20.5			.479	.344				
20239	do. ^a	26.50						6.80		4.0	5.0	24			.355	.299				
20611	do. ^b	71.87						25.32		+58.8	+25.0	21	7.71	23.13					Benzoic acid	
21049	do. ^c	35.00			0.211		23.86	.90	30.76	8.6	9.8	22			.345	.170	0.044	0.009	Salicylic acid	
19892	Blackberries	28.20					None.			8.1	8.1	20			.423	.192				
20249	Blueberries	12.18					None.			3.1	3.1	20.5			.166	.134				
19885	Cherries ^a	59.32					23.02			+13.0	12.4	24			.150	.152				Red coal tar.
20195	do. ^a	25.02					None.			5.4	5.2	24			.318	.260				
20240	do. ^a	24.81					2.26			.8	3.8	24			.363	.311				
20242	do. ^a	31.26					None.			6.8	6.8	24			.371	.275				
20243	do. ^a	63.90					11.36			.6	15.6	24			.187	.170				
20248	do.	50.88				.823	1.88			12.5	15.0	20			.361	.237				
20546	do. ^b	70.82			.494	.151	35.23	22.70	57.93	+48.8	+18.6	22.5		Present.	.198	.167	.032	.070		
19895	do.	47.37				.702	.75			13.0	14.0	20			.338	.232				
19898	do.	12.23				.401	1.04			1.4	2.8	21			.408	.283				
20136	do.	27.11					2.00			4.9	7.6	19			.321	.228				
20138	do.	24.73					2.37			2.2	5.4	19.5			.313	.214				
20230	do. ^b	68.86	1.65	.594			17.45			+65.2	+42.2	24.5	8.14	24.42	.334	.248	.027	.001		
20834	do. ^b	26.41					2.88			1.5	5.3	24.5			.395		Trace.			
21035	do. ^b	31.10					5.40			.0	7.2	22			.179	.100	.047	.009		Do.
21052	do. ^b	33.57				.058	.98			+2.3	+1.0	22	None.		.048	.017	.068			
21053	do. ^b	43.13					.60			11.1	11.9	24.5	None.		.045	.022	Trace.		Salicylic acid	Do.
21079	do. ^b	67.05				.215	12.75			+61.0	+44.0	21.5	3.68	11.04	.216	.114	.072	Trace.		

21085	do. ^b	46.01	.169	17.59	+ 9.3	-14.1	22	.186	.991	.012 Trace.
19897	Figs ^c	63.63		41.34	+51.4	- 4.2	19	.236	.198	
21086	do. ^b	56.98	.088	23.83	+14.1	-17.6	22	.131	.081 .064	.016
20231	Grapes		.303		- 5.0	- 5.0	20	.260	.147	
21088	Oranges ^c	62.70		15.00	+15.1	- 4.8	20	.468	.245	
19896	Pineapples	29.67	.563	None.	- 8.0	- 8.1	21	.462	.411	
20135	do.	21.90		5.02	.0	- 6.7	21.5	.318	.267	
20143	do.	23.81		1.11	- 5.0	- 6.5	19	.292	.196	
20234	do.	20.73	.509	1.05	- 4.4	- 5.8	21	.319	.243	
20228	do. ^a	38.60		6.50	+ .4	- 8.2	24	.059	.060	
20612	do. ^b	59.94		17.45	+55.6	+32.6	24.5	16.80	.629 .457	.092 .017 Benzoic acid.
19894	Plums	22.82	.769							
20139	do.	26.74	.710	.37	- 5.90	- 6.40	20	.196	.155	
21250	do. ^b	21.90	.730	.23	.00	- .30	24.5	.236	.161 .023	.002
20142	do.	36.40		.44	- 9.4	-10.0	20	.281	.215	
20238	do.	33.30	.602	.52	- 8.7	- 9.4	20	.234	.176	
20836	do. ^b	26.99		6.46	+ 1.6	- 7.0	22	.156	.110 .032	.023
21047	do. ^b	41.07		.60	-11.0	-11.8	21	.211	.104 .054	Trace.
20193	Peaches ^a	77.89		46.40	+68.0	+ 5.6	19	Present.	.299 .268	
19866	do.	18.75		2.52	- 1.50	- 4.90	19	.332	.239	
19871	do.	16.78		4.63	+ 1.20	- 5.00	20.5	.338	.239	
19893	do.	20.39	.485	3.82	- 1.00	- 6.20	21	.346	.268	
20233	do.	21.88	.303	7.31	+ 3.20	- 6.60	21.5	.351	.228	
21046	do.	27.20		.68	- 7.30	- 8.20	21.5	.253	.171 .089	Trace.
20140	Pears	27.14	.137	5.46	- 3.30	-10.50	21	.189	.140	
20144	do.	14.10	.161	.97	- 6.50	- 7.80	21	.249	.183	
20832	do. ^b	31.86	.110	6.59	+10.00	- 7.80	24.5	.151	.145 .028	Trace.
19870	do.	14.22		.96	- 4.0	- 5.3	19	.165	.097	
19872	do.	16.02		1.93	- 5.0	- 7.6	19	.230	.157	
19899	do.	16.05		5.31	- 1.4	- 8.5	21	.182	.114	
20134	do.	11.36	.205	1.72	- 4.0	- 6.3	20	.170	.116	
20137	do.	13.31		3.27	- 2.4	- 6.8	19	.210	.149	
20235	do.	29.21		10.94	+ 2.1	-12.5	21	.180	.141	
20609	do. ^b	60.94		27.26	+66.4	+30.5	24.5	.207	.157	.009 Benzoic acid.

the American samples put up in glass.

^b Foreign samples put up in glass.

• Contains a small amount of glucose.

^dStone jar.

TABLE 36.—*Composition of canned fruits*—Continued.

Serial number.	Description of sample. ^a	Total solids.		Per cent insoluble solids.	Per cent protein (N×6.25).	Acidity expressed as per cent H ₂ SO ₄ .	Sugars.			Polarizations.			Per cent dextrin.	Approximate per cent glucose (dextrin×3).	Per cent total ash.	Alkalinity as per cent K ₂ CO ₃ .	Sulphates as per cent potassium sulphate.	Chlorids as per cent sodium chlorid.	Preservatives.	Foreign coloring matter.
		Per cent in entire contents	Per cent in sirup.				Per cent reducing sugar.	Per cent cane sugar.	Per cent total sugar.	Direct.	Invert.	Temperature, degrees C.								
21048	Pears ^a		41.46					.90		-12.5	-13.7	21			0.081	0.052	0.065	0.008		
19875	do. ^a		8.95					.37		-4.0	-4.5	21			.139	.087			Saccharin.....	
21051	Raspberries ^a	34.36		3.45	0.562	0.656		.60		-7.8	-8.6	21			.308	.280	.051	.021	Salicylic acid.....	
21078	do. ^a	69.12		3.06		.43		3.34		+60.00	+55.60	24.5	10.16	30.48	.305	.252	.038	.001		Red coal tar.
20191	do. ^b		37.53					1.06		-8.0	-9.4	24			.279	.206				
20229	Strawberries ^a	71.76		1.28	.350	.205	45.76	19.08	64.84	+22.6	-2.8	22	2.54	7.62	.177	.099	.081	.021		Do.
20544	do. ^a	69.41		2.87		.37		6.53		+26.0	+17.4	24.5	4.81	14.43	.345	.281	.024	.003		Cochineal.
20610	do. ^a	68.82		3.96	.700	.419	51.67	7.13	58.80	+15.0	+5.5	22		Present.	.356	.210	.055	.009		Red coal tar.
20953	do. ^a	67.85		3.47	.562	.345	52.13	6.60	58.73	+18.4	+9.6	22		Present.	.355	.220	.090	.022		Do.
21045	do. ^a	48.72		2.00	.385	.240		.60		-11.2	-12.0	24.5	None.		.169	.136	Trace.	Trace.	Salicylic acid.....	Cochineal.
21050	do. ^a	31.86			.456	.563	29.28	.45	29.73	-8.0	-8.6	22		Present.	.385	.250	.047	.026	do.....	
19891	Cape Cod cranberry sauce.	64.86				.455		.62		+92.0	+91.2	20			.248	.158			Benzoic acid.....	
20247	Pickled figs ^c	59.56						26.96		+32.2	-3.4	24			.264	.232				
20164	Pickled water-melon. ^b				.137			20.60		+17.6	-10.0	22								
20545	Melange ^a	62.29						12.92		+7.4	-9.8	22			.184	.120	.072	.023		Do.
20244	Peach chutney ^b	44.67			.775			.80		-10.6	-11.0	20	None.		1.052	.520				

^a Foreign samples put up in glass.^b American samples put up in glass.^c Stone jar.

Table 36 gives the results of analysis of 74 samples representative of this class of goods. Of this number, 39 were put up in glass, 1 in a stone jar, and the remaining 34 in tin. Of the 39 samples put up in glass, 28 were of foreign origin. All of the tinned goods were of American manufacture.

A large number of the analyses given here consist merely in the examination of the sirup in which the fruit was put up, as this is sufficient to determine the presence or absence of glucose, preservatives, artificial sweetening materials, and, except in a few cases where the fruit alone was colored, of coloring matter, as well as the density of sirup employed.

A good idea of the quality of the fruit used for canning may be obtained from the general appearance of the product. Small, immature, and defective fruit is easily distinguished from the better goods. The quality of fruit governs, to some extent, the manner of its preparation. The lowest grade is commonly canned without the addition of sugar, and is always preserved in tin. A cheap but wholesome article is thus provided, for which there is great demand; at the same time the value of these goods will only warrant their preservation in the cheapest manner possible.

As an illustration of this class of goods may be mentioned "pie peaches," very small and imperfect fruit put up in water, after the removal of the pit, but without peeling. In this connection may also be mentioned the article prepared by soaking and canning dried fruit. This grade of goods is far inferior to canned fresh fruit, but finds a ready sale, at a relatively low price, in bad fruit years.

The grade of product prepared from a given quality of fruit depends largely on the amount of sugar added, or, in other words, the density of sirup employed. Generally speaking, the heaviest sirup is used with the best fruit, but good fruit is put up in sirup of all degrees of concentration, and even in water, according to the abundance of fresh fruit and the demands of commerce.

TABLE 37.—*Composition of fruits canned with glucose sirup.*

Serial number.	Description of sample.	Per cent total solids in entire contents.	Per cent total solids in sirup.	Per cent insoluble solids.	Per cent reducing sugars.	Per cent cane sugar.	Approximate per cent glucose.	Per cent total ash.	Alkalinity of ash as per cent K_2CO_3 .	Preservatives.	Coloring matter.
20611	Apricots		71.87			25.32	23.13			Benzoic acid.	
20546	Cherries	70.82			35.23	22.70	Present.	0.198	0.167		
20230	do	68.86	1.65			17.45	24.42	.334	.248		
21079	do	67.05				12.75	11.04	.216	.114		
19891	Cranberries....	64.34				.62	Present.	.248	.158	Benzoic acid.	
19897	Figs		63.53			41.34		.236	.199		
21088	Oranges		62.70			15.00		.468	.245		
20193	Peaches	77.89				46.40	Present.	.299	.268	Benzoic acid.	
20609	Pears		60.94			27.26	19.95	.207	.150	do ...	
20612	Pineapples		59.94			17.46	16.80	.629	.457	do ...	
21078	Raspberries ...	69.12		3.06		3.34	30.48	.305	.252		Coal-tar dye.
20229	Strawberries...	71.76		1.28	45.76	19.08	7.62	.177	.099		Do.
20544	do	69.41		2.87		6.53	14.43	.345	.281		Cochineal.
20610	do	68.82		3.96	51.67	7.13	Present.	.356	.210		Coal-tar dye.
20953	do	67.85		3.47	52.13	6.60	Present.	.355	.220		Do.

Table 37 gives the results of analysis of goods of this class that contain glucose. The samples containing glucose constitute but 17.6 per cent of the number examined—much less than in either jams or jellies. With one exception, the amount of glucose is below 30 per cent. Sample 19891 indicates from the polarizations that no cane sugar was used in its preparation. By reference to this table it is seen that all the glucose samples have a content of solids of 60 per cent or more, while Table 36 shows that only 8 samples having more than 50 per cent of solids are free from glucose. This condition seems to bear out the impression that glucose is very commonly employed where its substitution for the more expensive sugars can be made of financial interest to the manufacturer, hence the limited use of glucose in the whole-fruit product is due to the fact that in many cases no sugar is added, while in others the amount used is not sufficient to warrant adulteration.

The table following shows the amount and nature of adulterants found in canned fruit products examined, together with the sources of the samples.

TABLE 38.

	American foods.	Foreign foods.	Total.
Total number of samples.....	46	28	74
Number of samples containing glucose.....	4	11	15
Number of samples containing preservative.....	3	8	11
Number of samples containing foreign coloring matter.....	1	9	10
Number of samples containing more than one adulterant.....	1	10	11
Number of samples adulterated.....	6	18	24

The use of coloring matter was confined to the fruits put up in glass. Of six samples of strawberries examined but one was free from artificial coloring matter. Coloring matter is probably most frequently used with this fruit, as it so readily loses its natural color after canning. But two samples of goods preserved in tin contained any form of preservative, and in one case the material used was saccharin, which performs the double function of preservative and sweetening agent.

The addition of preservatives to fruit put up in hermetically sealed packages and sterilized is unnecessary. In some classes of foods it is advantageous to preserve the product in a partially prepared condition for a considerable length of time, and complete the process in a much smaller plant than would otherwise be required. This is not customary, however, in the preparation of canned fruit. The only condition, therefore, that would call for preservatives is the use of imperfectly sealed receptacles or the lack of complete sterilization. The difficulty of hermetically sealing glass receptacles, especially those with a wide mouth, is well known, and the problem is made more complex when the goods are shipped to a great distance.

The customs of different firms differ so widely regarding the relative character of fruit packed in tin and glass that generalizations are difficult. It may be said, however, that the lowest grade of products is preserved in tin. Pie peaches, restaurant goods, and soaked goods (canned dried fruit) are prepared for a market that demands cheap products. It is necessary to choose the least expensive package available. In addition to this the appearance of these articles would not be inviting in glass.

In high-grade fruits, on the other hand, some canners place exactly the same products in glass and tin. Again, the expense of shipping bottled goods, both on account of breakage and freight rates, practically prohibits the preservation in glass of even high-grade goods that are to be shipped to a great distance, and many firms pack all their fruit in tin even for local markets. The average quality of tinned fruit is inferior to that preserved in bottles, and the lowest quality of the former is far below that of the latter.

BRANDIED FRUITS.

The brandied goods, aside from their content of brandy, are very similar to the canned fruits. While a number of samples were found upon the market, it is probable that the demand for this class of goods is not large. Seven samples were examined, of which five were imported. No adulterants were detected in any of these samples. Three were put up without the addition of cane sugar. The others contained amounts varying from 5.84 per cent to 21.20 per cent. Alcohol was determined on four of the samples, and was present in sufficient amount to prevent any fermentation.

TABLE 39.—*Description of brandied fruits.*

Serial number.	Manufacturer.	From whom purchased.	Claims of manufacturers.
20162	Gordon & Dilworth, New York...	C. C. Bryan, Washington, D.C....	Brandied cherries.
21033	A. Jourde, Bordeaux.....	Popovich & Abramovich, New Orleans, La.	Cherries mirabells.
21058	Marx, Bordeaux.....	J. G. Swarbrick, 321 Camp street, New Orleans, La.	Cerises à l'eau-de-vie.
20192	G. W. Dunbar & Sons, New Orleans, La.	N. W. Burchell, Washington, D.C..	Fig preserves.
19810	A. Azema.....	U. S. appraiser, Port of New York.	Peaches in brandy.
21034	A. Jourde, Bordeaux.....	Popovich & Abramovich, New Orleans, La.	Peches à l'eau de vie.
21032	do.....	do.....	Do.

TABLE 40.—*Composition of brandied fruit.*

Serial number.	Description.	Per cent total solids.	Per cent total ash.	Per cent alcohol.	Per cent cane sugar.	Polarization.			Alkalinity of ash as potassium carbonate.	Acidity calculated to per cent H ₂ SO ₄ .	Sulphates as potassium sulphate.	Chlorids as sodium chloride.
						Direct.	Invert.	Temperature, degrees C.				
20162	Cherries.....				10.90	+ 8.40	-6.00	24				
21033	do.....	20.70		16.60	.30	- 1.00	-1.40	25				
21058	do.....	11.20		17.70	None.	- .40	- .50	25				
20192	Figs.....	38.36	0.274		21.20	+26.00	-2.00	24	0.209			
19810	Peaches.....	5.42	.210		5.84	- 1.00	-8.80	21	.129	0.159	0.055	Trace.
21034	do.....	15.10		16.10	7.18	+ 6.20	-3.00	25				
21032	Prunes.....	20.80		16.60	None.	- 1.30	-1.40	25				

FRUIT BUTTER.

The limited number of fruit butters found upon the market indicates that this class of fruit products in small receptacles does not

have an extensive sale. Of the three samples examined, one was peach and the other two apple, and only one of these (No. 20160) is, in the strict interpretation of the term, a fruit butter. In the manufacture of this product as ordinarily produced outside of the factory, the juice is concentrated and then boiled with sufficient amount of the fruit to give it the desired consistency. The product should be tart in flavor and thick, but not jelly-like, in consistency. No. 20141 is a peach-butter compound, said to contain 50 per cent peach, 30 per cent glucose, and 20 per cent cane sugar. Only a small amount of cane sugar was found, and it is possible that that originally present might have been nearly completely inverted, since these goods are usually boiled for a considerable length of time. No. 20160 contained but a very small amount of cane sugar, which might have been normally present in the apple; and the composition of this sample shows it to be typical of this class of fruit products. No. 20237 contains glucose, and the high polarizations indicate that no cane sugar had been added in the process of manufacture.

TABLE 41.—*Description of fruit butter.*

Serial number.	Manufacturer	From whom purchased	Claims of manufacturer.
	<i>Fruit butter.</i>		
20141	Williams Bros. & Charbonneau, Detroit.	Henry Barge, New York	Peach-butter compound: 50 per cent peach, 30 per cent glucose, 20 per cent granulated sugar.
20160	H. J. Heinz Co., Pittsburg, Pa.	do	Heinz's apple butter, Keystone brand.
20237	West Virginia Preserving Co., Wheeling.	Park & Tilford, New York	Fort Henry brand, apple butter.

TABLE 42.—*Composition of fruit butter.*

Serial number.	Description.	Per cent total solids.	Per cent total ash.	Per cent cane sugar.	Polarization.			Alkalinity of ash.	Per cent of protein (N $\times$ 6.25).	Acidity calculated to per cent H ₂ SO ₄ .	Preservative.
					Direct.	Invert.	Temperature.				
20141	Peach butter	43.12	1.127	0.89	+33.2	+32.0	19	0.680			
20160	Apple butter (in stone jar) .	47.91	.703	.75	-12.0	-13.0	20	.448	0.350		Benzoic acid.
20237	Apple butter *	37.55	.463	.30	+54.4	+54.0	21	.176		0.441	Do.

* See correspondence with manufacturers, p. 102.

SOLID MARMALADES.

Three samples of solid marmalades were examined. These were secured in Mexico by Dr. E. Palmer, of the Division of Botany. Two were prickly-pear marmalade, the third was quince. Nos. 22294 and 22295 were made without the addition of cane sugar, and this fact accounts for the high content of ash, reducing sugars and protein. Of particular interest in the analysis of these two samples is the positive polarization after inversion. This may be due to an excess of dextrose, or, as was indicated in the discussion of the data in Table 18, the processes of heating might have converted some of the otherwise insoluble constituents into soluble optically active bodies. No. 22296, the quince marmalade, contained a large amount of cane sugar, and hence can not be compared with the two samples of prickly-pear marmalade. It is probable that this class of fruit preserves finds little, if any, sale in the markets in the States.

TABLE 43.—*Composition of solid marmalades.*

Serial number	Description.	Per cent total solids.	Per cent total ash.	Per cent reducing sugar.	Per cent cane sugar.	Per cent total sugar.	Polarization.			Alcohol precipitate.	Per cent of protein (N×6.25).
							Direct.	Invert.	Temperature, degrees C.		
22294	Prickly-pear preserve.....	87.74	1.76	77.18	1.37	78.55	+ 8.4	+ 6.6	24.5	5.37	2.437
22295	do.....	87.48	1.92	73.98	.76	74.74	+ 5.8	+ 4.8	24.5	5.96	2.687
22296	Quince preserve.....	91.93	.577	23.50	56.15	79.65	+47.0	—27	24.5	8.30	.487

CORRESPONDENCE WITH MANUFACTURERS.

In all cases where results were obtained that might in any way be considered unfavorable to the manufacturer of the goods these results have been reported to the manufacturer previous to their publication in order to give an opportunity for such explanation as might be considered pertinent. The substance of the correspondence of the manufacturers upon various points that have arisen in regard to the analyses reported in this bulletin is given below.

It must be remembered that in purchasing goods upon the market it is not possible to always obtain samples representative of the product that is being put out at the time the goods are purchased. The methods of manufacturers frequently change in reference to the ingredients employed, and goods may remain some length of time upon the grocers' shelves. The difficulty encountered in obtaining fresh prod-

ucts is apparent. This fact accounts for many of the objections that have been offered to the results obtained in the analyses.

Generally each manufacturer puts upon the market more than one grade of goods, the number depending upon the demands of the trade. In making a miscellaneous collection of samples it is not probable that all these grades have been obtained, since no special effort has been made to secure all the grades of each manufacturer.

It frequently occurs that only the cheaper grades of goods were obtained, hence the results reported are not always representative of the entire output of each manufacturer. The explanations offered by the manufacturers likewise apply frequently to their cheaper grades rather than to their products as a whole. (See introduction, p. 3.)

The correspondence follows:

I am in receipt of your favor addressed to Alden & Nicholson giving some tests you have made on some preserves and jams made by them. This firm has been out of existence for some time, and had no successors. The goods you tested were made some time ago, and as no goods are now made under the name of Alden & Nicholson in any way, there should be only isolated cases where parties had not disposed of their stocks on hand at time of dissolution of the company. I thank you for the courtesy shown in your letter.

Yours, truly,

C. S. ALDEN.

NOTE.—This communication is one of several which illustrate the fact that the goods found on the market are not always representative of the output of manufacturers at that time. A failure to appreciate this fact might readily lead to unjust criticism of manufacturers, and to controversies between them and control laboratories.

We have yours of the 11th instant to hand, giving us results of analysis on jelly (current flavor and peach flavor), serial numbers 20216 and 20217, also on raspberry jam, serial number 19969, and strawberry jam, serial number 19968. In reply we would say that we * * * find numbers 20216 and 20217 correct, with the exception of the mention of starch being present; as far as the employment of same is concerned by us in the manufacture of jelly. A very small per cent of glucose is used in order to prevent it from crystallizing, but never any starch in any jams or jelly. If present, then it must appear in the fruit employed itself. We note further that no sugar is found in the raspberry or strawberry jam, while the fact is that 50 per cent of granulated sugar is used in the berries themselves at the time of preparing the fruit when in season. When these berries are made in low-grade jams a jelly of apple juice and glucose is made and the berries boiled and mixed therein. Whether all the granulated sugar used could be turned into invert sugar through two processes of boiling we know not, but such are absolutely the facts as to our method of making the lowest grade of strawberry, raspberry, or other jams. * * *

THE AMERICAN PRESERVE COMPANY.
LEWIS J. LINK, *President*.

NOTE.—The samples of raspberry jam above mentioned contained no cane sugar, and the strawberry jam contained only 1.19 per cent. According to the method of manufacture reported for these jams it is probable that the addition of the mixture of fresh berries and cane

sugar was comparatively small, and the amount of cane sugar thus added could very easily have been completely inverted in the process of preparation. For the effect of the preserving process on cane sugar, see p. 52.

We have your circular of the 11th with reference to your bulletin giving the results of examination of fruit products. * * *

In reference to starch, we have to say that * * * it is not our custom to use starch or any farinaceous adulterative in fruit jams or preserves. We think the tendency * * * is to substitute apples in the place of so much glucose, for the reason that it makes a better jam in many ways.

In regard to the use of preservatives in fruit jams, we consider it a waste of money. If the goods are properly made we don't think a preservative is necessary. * * * There is, however, a certain line of very high grade preserved fruits that seem to be demanded by the people in good circumstances, in which it is necessary to use a very small quantity of preservative. This is owing to the fact that the goods are demanded in glass. They are entirely filled with sugar and sunk in a sugar sirup.

These goods at this time in the year will keep, but in the Southern climate or during hot weather they will not keep.

This is entirely owing to the fact that no manufacturer has been able to find a glass package that can be hermetically sealed and stand transportation. If this package could be found it would cure a whole lot of these difficulties.

ANDERSON FOOD COMPANY.

Your favor of the 11th instant, giving the results of your analysis of our grape fruitate, guava jam, and pickled figs received. We thank you for submitting same.

* * * We aim to put up pure goods, but in the fruits that are of a red color, namely, berries, guava, and currants, we use some red coloring to make the appearance better. We have for years used ——— "sugar red color," which we understood passed the pure-food laws of Ohio, and which Mr. ——— (the manufacturer) assured us was purely vegetable color; hence we are surprised that your analysis shows coal-tar dye in our guava jam. We do not put coloring matter in to cheapen the goods in in any manner, but only to aid the appearance. We are trying to make an aim on our goods as pure foods. * * * Regarding the samples of grape fruitate, it is impossible for us to account for any benzoic acid being in same unless it could have been one of the first few batches we made. In this we put a small amount of preservaline No. 1. Since that time, which was over two years ago, we have not used any antiseptic whatever, and if this sample contains benzoic acid it must have been some of the first lot that went into Washington.

BISHOP & Co.

NOTE.—Two additional samples of grape fruitate (probably old stock) bought at Washington, D. C., also contained benzoic acid. Its presence is doubtless due to the use of the commercial preparation mentioned above. A sample of the above-named color furnished by Bishop & Co. was found to be of coal-tar origin, hence the color was misrepresented by the manufacturer when sold as a vegetable color.

Replying to your results of analysis under date of November 11 to the Campbell Preserve Company and the Crescent Preserving Company, we would state that * * * your results are relatively correct; that is to say, in the Crescent goods the percentage of glucose is twice as much as sugar. The preservative used in all instances is sodium benzoate, not benzoic acid in its literal sense.

JOS. CAMPBELL PRESERVE CO.
ARTHUR DORRANCE, *President*.

Replying to your circular letter of the 11th, and in reference to the examination of various items of our product, would say that we * * * feel warranted in making the following general statement: During the past three years we have used no glucose whatever in the jelly that we have made. In the same period of time we have used no salicylic acid either in jelly or jam. * * * If your examinations, as shown by your report, have been carefully made, we are at a loss to understand the occasion that would warrant the findings unless you have gotten hold of extremely old items produced by us, and even then in some instances we can not understand the occasion of the reports being as you state. We feel that in all fairness to us, analyses should be made of new goods or those of more recent production. * * * After discontinuing the use of salicylic, we may have had up to the next season some goods that had been prepared with it, but in reconditioning these we used no more salicylic, but attempted to volatilize this, and then used benzoate for the direct preservative.

CURTICE BROTHERS COMPANY.

Your letter of the 11th instant received, and we note all you have to say. We use no starch whatever in any of our product; if it shows any starch it must come through the glucose. In the item of jam No. 20227 we note that you show no sugar; there is about 10 per cent granulated sugar in our cheap jams. We have discontinued using benzoate of soda in our jams, as we knew that the Pennsylvania food law had ruled against it.

THE E. G. DAILEY COMPANY.

We are in receipt of your circular letter of the 11th, and we note your report on analysis No. 20237 of our Fort Henry brand apple butter. We suppose, of course, that your report is correct, excepting it does not show the percentage of sugar. We use a certain percentage of sugar in this apple butter, but do not use starch in any of our fruit products.

GEO. K. McMECHEN & SON.

We are in receipt of your favor of the 11th instant advising us of the result of an analysis made of our apple jelly, which was presumably bought in open market, as we have no record in our office of having sent you any.

As stated by you, there is no comment to make, except to explain the presence of starch, which evidently is in so small quantity that no record was made of its percentage. This starch is undoubtedly due to the character of the apples used (not as a thickener), as we have made no change in our recipe for several years, or, possibly, to imperfect filtering of the apple juice, or both. We have endeavored to overcome this (only) objection, but have not succeeded so far. We shall continue our efforts, however, to remedy this phenomenon.

DODSON-BRAUN MFG. CO.

In reply to your letter of November 11, would say that the analysis given our products is undoubtedly correct in every case, and excepting the strawberry jam, No. 20182, entirely satisfactory to us. * * * We have never put salicylic acid into strawberry jam except in one instance, and it evidently so happens that your sample has been selected from this particular lot. Last season we became sold out of strawberry jam, and to supply the demand we used some berries which had been barreled with sugar and preservaline and carried until midwinter. This is the only case in forty years of packing in which we have done this, and it will perhaps not be necessary again in the next forty years. It is much more satisfactory to make the jam from fresh berries, and certainly does not need any preservative in making. We can only say that we regret that the sample which you happened to get must have been taken from this lot.

ONEIDA COMMUNITY, Limited.

We have your favor of the 11th instant and note contents. * * * One item which strikes us as particularly incorrect is number 20218 (Table 30), which you call cherry jam XX brand. This is our best brand, and we take extraordinary pains with the goods. We use fresh fruit and granulated sugar only in the manufacture of these goods, and your analysis says 40 per cent glucose. We do not use starch for any of our preserves, jams, and jellies. * * * Undoubtedly you have obtained the samples which you analyzed from dealers in Washington. Washington trade buys principally the cheapest grade of goods we make. We sell only a small amount of such goods; we do not care to make and make no effort to sell them. We make six different grades of preserves, jams, and jellies, each grade differing from the other in quality and price. * * * Buying up a lot of samples in a market where a large percentage of low-grade goods is used, making an analysis of these samples and publishing the result as indicative of the class of products we make, would be doing us a great injustice. It would place us among a class of manufacturers to which we do not belong.

P. J. RITTER CONSERVE COMPANY.

I desire to thank you for giving me an opportunity to correct your finding as relates to my present output of fruit jellies. We are closing our jelly in an improved manner these past two years or more, sealing it hermetically with paraffin, and thus obviating the necessity for any germicide to prevent mold. We use no starch as your letter, though not your analysis, would seem to imply. * * * In closing I beg to repeat that we are now using no salicylic acid, and think your sample must have been of a date of several years past.

Very truly,

Mrs. SARAH C. STONE.

The jellies analyzed by you were prepared after an old formula, which was used by a former superintendent of our factory in this city, but which has been abandoned by our present superintendent, and the goods now being put up under the same label would show entirely different results by analysis, and we are informed that no preservative is being used. We do not know that your Department objects to the use of certain preservatives, but we have abandoned the use of them in the preparation of all such articles as could be safely put up and sold to dealers in all sections of our country without.

SPRAGUE, WARNER & Co.

Your letter of the 11th received giving us analysis of package of blackberry jam. We think the analysis you have made is all right excepting the salicylic acid. This must be a package of fruit put up about two years ago, as we have not used any salicylic acid as a preservative for the last two years. We have been using benzoate of soda since we abandoned the use of salicylic acid, as we find that this is equally as good a preservative and the food commissioners have been allowing the use of this preservative. We do not put any starch in any of our products, but we use considerable glucose, also apples, for a basis of cheaper grades of jams and preserves.

THE J. WELLER COMPANY.

Replying to your favor of the 11th, * * * we note in No. 20147 blackberry jam your analysis indicates 34.08 per cent of sugar, while none of the other samples indicate sugar. The fact is, we use the same quantities of sugar in all the different fruits, the formula being made up of so much sugar, fruit, and glucose, and some apple stock. These are compound goods, sold at low prices to meet the wants of the masses of the people who can not afford to pay the high price that the jams composed entirely of fruits and cane sugar would demand. We put out our best quality of jams under the Dragon brand, and in these we use nothing but sugar and fruit. We can assure you we use no starch whatever, so the starch must come from the fruits and glucose. And in this connection would say we do not use a pound of starch in our factory as a thickener.

THE WILLIAMS BROTHERS COMPANY.

PART II.—MICROSCOPICAL EXAMINATIONS OF FRUITS AND FRUIT PRODUCTS.

By BURTON J. HOWARD.

The close structural similarity of many of the fruits used in canning makes the problem of the identification of the constituents by no means easy. Apple pulp, which is used as a filler in many fruit products, is so devoid of characteristic structure that any method giving hints as to its presence is of value. It is a well-known fact that starch is usually present in most fruits during some periods of growth, but that in many it more or less completely disappears when the fruit is ripe. With this in mind, some examinations were made of apples, pears, peaches, quinces, grapes, raspberries (red and black), strawberries, and tomatoes, in various stages of ripening. Apples more than any other fruit are used in an immature state, and starch can often be detected in fruit products composed wholly or in part of apples. Concerning the morphological features of apple starch it may be said that it consists of both simple and compound grains. The simple grains are globular in form, and according to Browne^a are about 9 microns in size. This size, however, we have found to vary greatly, since in maturing the grains are converted into sugar and during this period decrease in size. In one specimen the average size was found to be 9 (3.5 to 14) microns, while in another it was 11.8 (7 to 17.5) microns. This fact of change in size was strikingly shown in the case of two Kieffer pears. In one which was nearly ripe the average was 4 (1.5 to 5) microns, while in a green specimen the size was 13 (3 to 29) microns. The compound grains, which are very abundant in green fruit, are composed of 2 to 5 grains attached together, giving a clustered or tetrahedral effect. Such grains are seldom seen in samples of apple starch obtained by grating and sedimentation, since they are easily broken up into truncated simple grains in the process of grating. The grains are marked by a stellate or linear fissure or depression and some with a good light show faintly concentric rings. The grains are active toward polarized light, and show colors with the selenite plate. As the apple ripens, starch begins to disappear near the center of the fruit, and later at the periphery. The last traces are to be found in the cells adjacent to the fibrovascular bundles. Fully ripe apples contain no starch whatever except in bruised portions.

^a Penna. Dept. of Agric., Bull. 58.

From the above it is evident that in the examination of jams and similar products starch can best be detected in cells clinging to the fibro-vascular bundles, which may be separated as directed under "Technique" (p. 106). In the tomato the last traces are found in the placental region. In the strawberry it remains longest in the central part of the pulp or enlarged receptacle. As in the apple, portions of the fruit bruised before maturing retain the starch practically unchanged.

In green pears considerable starch is present, but it disappears at a comparatively earlier stage of ripening than in the apple. In peaches it seems to disappear still earlier, for only in specimens greener than would probably be used in canning was there an appreciable amount found. In the quinces, though firm and sound, only a faint trace was found October 8, while apples—Rambo, Northern Spy, Baldwin, Greening, Russet, and others—obtained at the same time from the same orchard in Michigan showed an abundance of starch.^a Although grapes were examined in various stages of maturing, from green to perfectly ripe, no starch was found. The same was true of the raspberries. It should be said here, however, that the grapes were gathered late in the season, after the first frosts, which may have hastened the disappearance of the starch had there been any in normal conditions. Strawberries, up to the time the flush of ripening appears, show a small amount of starch, but this seems to disappear before many of them would have been picked for market or canning. In the case of tomatoes the starch persists somewhat longer, but seems to disappear approximately with the last traces of green color, although the main portion is gone before this. Tomato starch, except on long boiling, does not swell up and become as mucilaginous as that of apple, but remains as little clots within the cells.

From the above it appears that we are able to assign the presence of starch in fruit products to any one or all of three causes, viz, (1) the use of immature or green fruit; (2) the use of apple pulp; (3) the addition of starch as such. It should be remembered here, however, that the absence of starch in no way precludes the possibility of apple pulp having been used. Although boiling tends to remove starch from the cells, as is readily seen in the case of many apple jellies, yet long-continued boiling is required to remove it so thoroughly from the cells that they will not show a greater starch content than the surrounding liquid when the iodine test is applied. Even in the case of apple butter more starch was found within than outside the cells. This fact may be used to advantage for detection of commercial starch in pulped products, such as jams, preserves, and marmalades; but it must be used with caution, since a small amount of starch might be added to products made from starch-containing fruits without detection. Even

^aAll of these varieties, with the exception of Rambo, are winter apples, some of them ripening late in the winter.

in apple butter, where the cells were well separated from each other, the starch was found to be much more abundant inside than outside the cells. In making this test a small amount of rather strong iodine solution (iodine, 1 gram; potassium iodide, .2 grams; water, 300 cc) should be used, else a considerable diluting effect will occur.

Adams^a gives Hoffman's violet (Dahlia), followed by iodine, as a color test for apple pulp, claiming that the stain for apple is very different than that obtained for other fruits. Although we have tried the aqueous and anilin water solution, the color has not been found sufficiently characteristic to be reliable in most cases. What at first seemed to be characteristic differences on closer work were found to be caused by differences in the sugar or acid content, which served to mask or vary the normal staining effect.

Malfatti^b has gone quite carefully into some of the anatomical characteristics of apple and pear, but as he confined his efforts largely to features of the epidermis, capsule walls, and seeds—parts usually removed in preserved goods—the differences are obviously of little value where the pulp alone is used. In the table at the end of this article the effort has been made to bring together in concise form the leading morphological and chemical characteristics of some of the fruits most like apple in the pulp condition to assist in the identification of apple when present.

A careful histological study of the seeds of those fruits in which the entire fruit is used will aid greatly in their identification. Marpmann^c has worked out certain histological features of the seeds of figs, raspberries, currants, gooseberries, whortleberries, blackberries, strawberries, and elderberries which characterize these species and assist in their identification. Since A. L. Winton, of the Connecticut Experiment Station, had already begun a careful histological study of various fruits, no further detailed work of that nature was undertaken in connection with this bulletin.

The writer became identified with the Bureau of Chemistry after the chemical work described in Part I was completed and the samples discarded. It was therefore impossible to apply these methods to those samples, as it was not deemed advisable to procure new samples of the same goods on account of the press of work in other fields.

PREPARATION OF SAMPLE.

To prepare the sample for microscopical examination a portion, after being warmed with several times its bulk of water, is shaken up sufficiently to free the particles from sugar and allowed to settle. If the sample is finely pulped the settling process can be greatly hastened

^aAnalyst, 1884, 9, 101.

^bZtschr. Nahr. Hyg. 1895, 10, 265, 297, 313, 329.

^cZtschr. aug. Mikros., II Bd., 4 Heft.

by using a centrifuge. By decanting the supernatant liquid and placing the final product in a large watch glass a macroscopic examination can be made, which will usually be found suggestive as to the probable components. Portions can then be mounted for microscopic examination.

TECHNIQUE OF EXAMINATION.

The examination for starch in cells is made by iodine in potassium iodide solution.

To examine the pulp cells a sample was fixed to the slide by evaporating to dryness at about 50°, or by smearing the slide with Mayer's albumen fixative before putting the sample upon it and then rinsing with 90 per cent alcohol before beginning the staining process. The former method, however, was found more satisfactory. For a temporary mount no clearing agent is used. For Adams's test the best results were obtained by using a solution of Grüber's dahlia, made up according to the usual formula (dry stain, 1 gram; anilin water, 80 cc; 95 per cent alcohol, 20 cc). A drop of stain was added and allowed to act one minute, rinsed in water carefully thirty seconds to remove excess stain, and then treated with iodine solution thirty seconds before covering. For permanent specimens Gram's gentian violet or anilin safranin (these stains are made up of dry stain, 1 gram; alcohol, 20 cc; anilin oil, 3 cc; water, 80 cc) gives good results when sections are mounted in balsam. If the material is stained two to three hours in the safranin and then after rinsing with water stained for one minute in gentian violet, the stone cells are differentiated from the pulp cells, since, being composed of lignified tissue, they take the safranin well, but very slowly the gentian violet which, on the other hand, stains the cellulose pulp cells very readily.

The scheme of identification outlined is quite largely based upon an examination of the components of the fibro-vascular bundles, which in many cases have characteristic elements. The sample for such examination is mounted in a drop of chloral hydrate (crystals 8 parts, water 5 parts) and heated for a few seconds to boiling, covered, and by gently tapping the cover glass the bundle is broken up into its elements. Marpmann^a recommends the use of alcoholic phenol as a clearing agent, but we have used chloral hydrate with better results, and it has the advantage of assisting in the maceration process.

A second method consists of staining with acetic acid-methyl green, and after withdrawing the excess of stain with a strip of filter paper and mounting in water, separating the component elements by tapping the cover glass as before. By this means the elements are easily distinguished, though they are not cleared, as in the chloral-hydrate method.

^a Ztschr. ange. Mikros., II. Bd., 4 Heft, p. 97.

In the set of drawings and photomicrographs herewith the microscopical characteristics of the fruits examined thus far are shown as far as possible.

Fruit.	Starch.	Histology.		Remarks.
		Stone cells.	Elements of fibro-vascular bundles.	
Apple ^a ...	May or may not be present.	Rarely elongated stone cells found in fibro-vascular bundles.	Spiral, annular, reticulated, and dotted vessels; occasional nearly smooth bast fibers.	
Pear ^b	Rarely present.	Abundant, with lumen nearly closed.	Similar to above; but bast fibers often intermediate in form between bast and stone cells.	
Quince ^b .	None found at time of preserving.	Abundant; lumen about $\frac{1}{2}$ diameter of cells.	Very similar to apple	Pulp cells oblong, and with iron perchlorid or ammonia molybdate give tannin reaction.
Peach ^ado.....	do.....	Modified elongated stone cells found.	No spiral vessels; mostly reticulated form.	Pulp cells vary in form from globular in pulpy portion to slender, spindle-shaped ones in vicinity of fibro-vascular bundles.
Apricot ^a .	None found....	None	Rarely spiral vessels; many greatly elongated, reticulated forms.	
Plum ^ado.....	do.....	Many stone-like cells in angles of fibro-vascular bundles.	Reticulated and spiral vessels.	Crystals in fibro-vascular bundles.
Grape ^a ...	None.....	None	Composed entirely of spiral and annular vessels.	Needle-like crystals; many small rectangular cells; often bunches of crystals attached to fibro-vascular bundles.

^a Adams's test for pulp cells: Pinkish purple.

^b Adams's test for pulp cells: Pinkish purple, with more of brick-red tint and color.

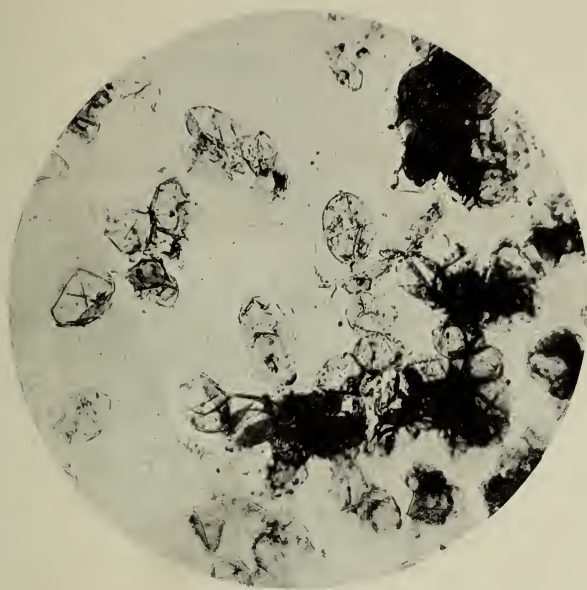


FIG. 1.—FRUIT-PULP CELLS, APPLE. $\times 30$.

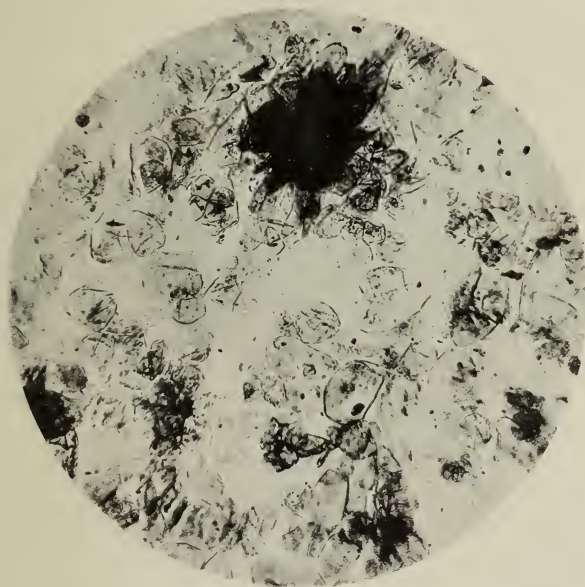


FIG. 2.—FRUIT-PULP CELLS, PEAR. $\times 30$.

Photographs by Burton J. Howard.

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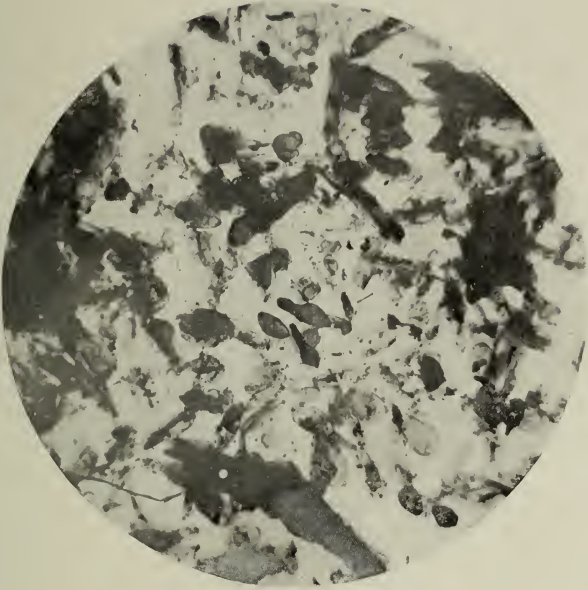


FIG. 1.—FRUIT-FULP CELLS, QUINCE. $\times 30$.

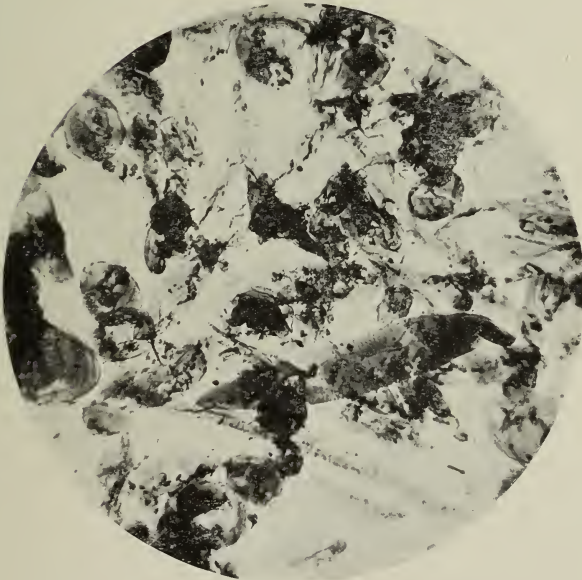


FIG. 2.—FRUIT-PULP CELLS, PLUM. $\times 30$.

Photographs by Burton J. Howard.

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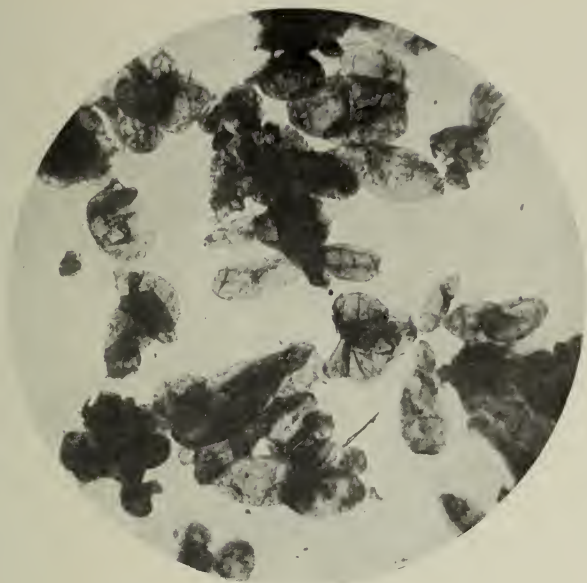


FIG. 1.—FRUIT-PULP CELLS, PEACH. $\times 30$.

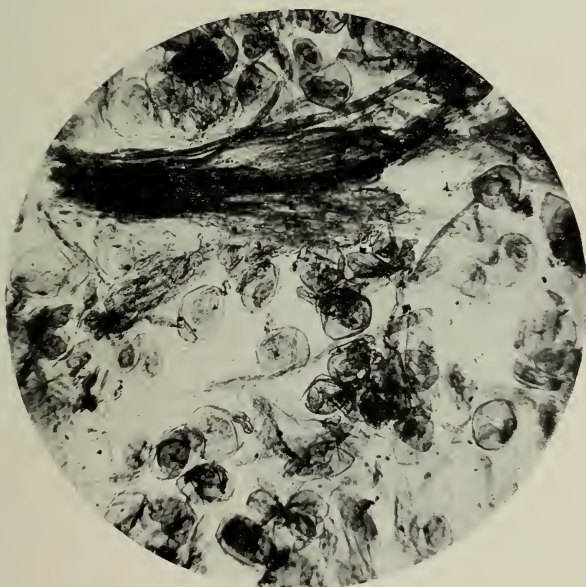


FIG. 2.—FRUIT-PULP CELLS, APRICOT. $\times 30$.

Photographs by Burton J. Howard.

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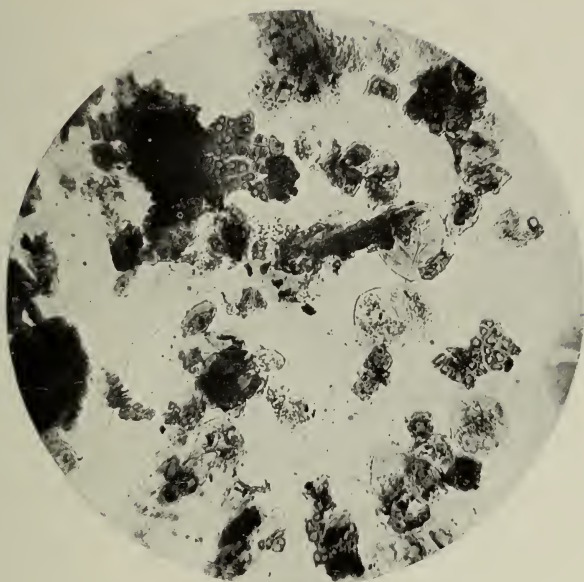


FIG. 1.—FRUIT-PULP CELLS, GRAPE. $\times 30$.

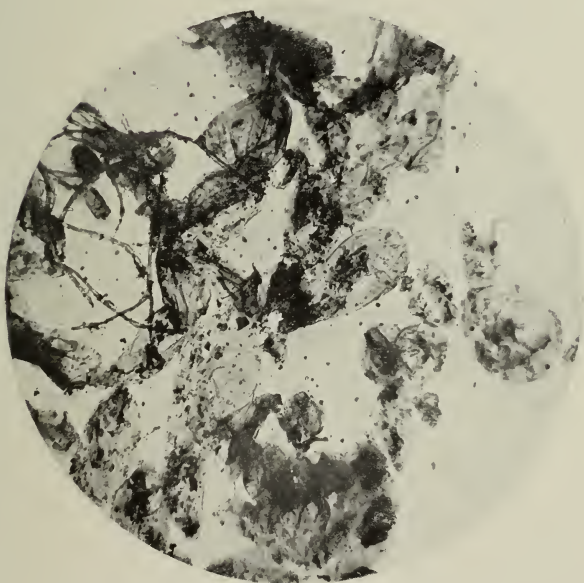


FIG. 2.—FRUIT-PULP CELLS, STRAWBERRY. $\times 30$.

Photographs by Burton J. Howard.

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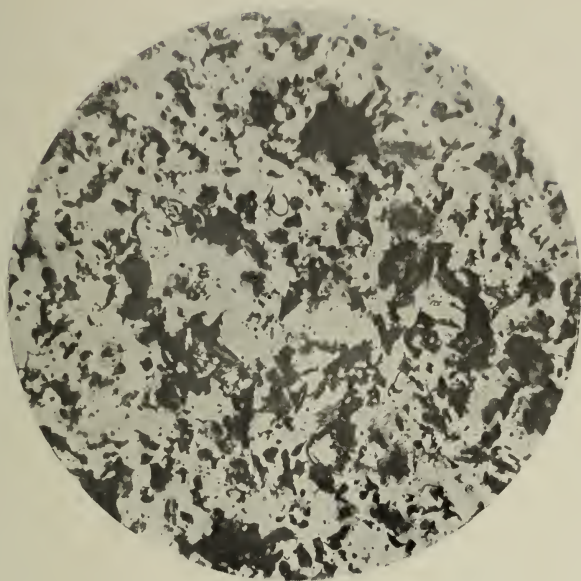


FIG. 1.—FRUIT-PULP CELLS, RED RASPBERRY. $\times 30$.

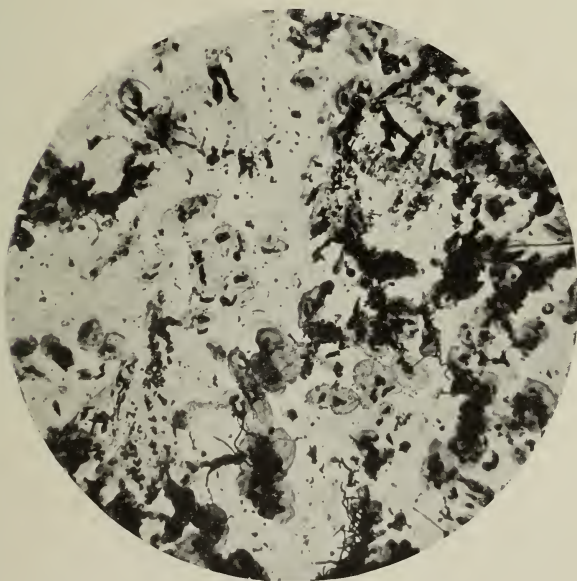


FIG. 2.—FRUIT-PULP CELLS, BLACK RASPBERRY. $\times 30$.

Photographs by Burton J. Howard.

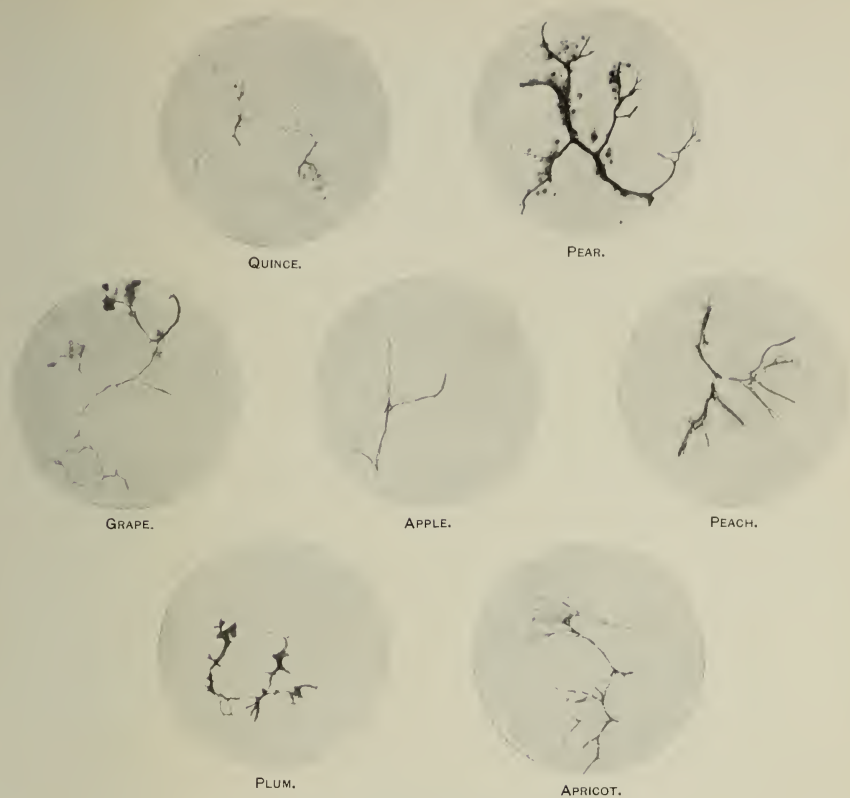


FIG. 1.—FRUIT FIBROVASCULAR BUNDLES. $\times 2$.

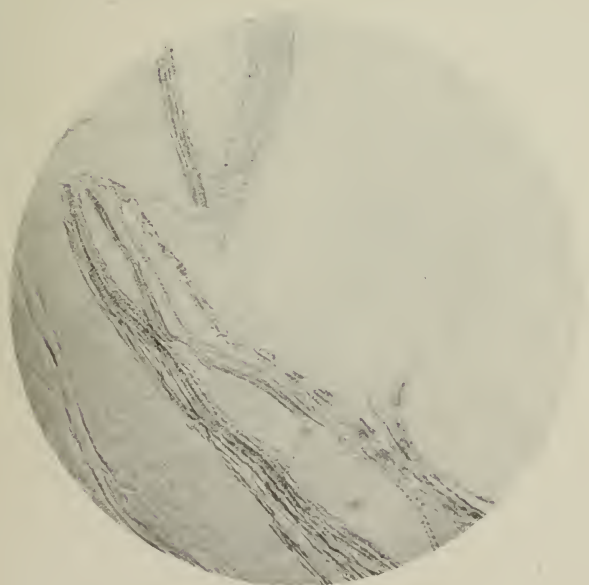


FIG. 2.—FIBROVASCULAR BUNDLES OF APPLE. $\times 75$.

Photographs by Burton J. Howard.



FIG. 1.—FIBROVASCULAR BUNDLES OF PEAR. $\times 75$.



FIG. 2.—FIBROVASCULAR BUNDLES OF QUINCE. $\times 75$.

Photographs by Burton J. Howard.

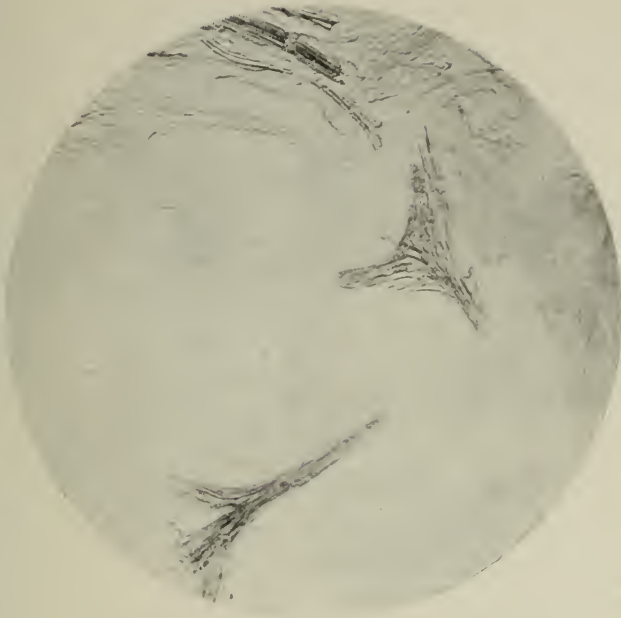


FIG. 1.—FIBROVASCULAR BUNDLES OF PLUM. $\times 75$.



FIG. 2.—FIBROVASCULAR BUNDLES OF PEACH. $\times 75$.

Photographs by Burton J. Howard.



FIG. 1.—FIBROVASCULAR BUNDLES OF APRICOT. $\times 75$.



FIG. 2.—FIBROVASCULAR BUNDLES OF GRAPE. $\times 75$.

Photographs by Burton J. Howard.

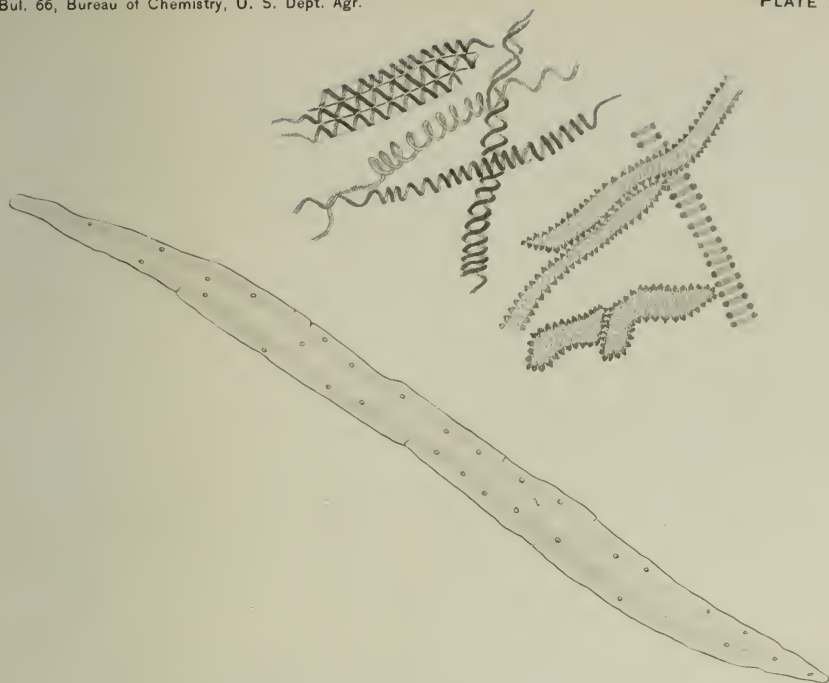


FIG. 1.—ELEMENTS OF FIBROVASCULAR BUNDLES OF APPLE. X 425.

DRAWN BY BURTON J. HOWARD.

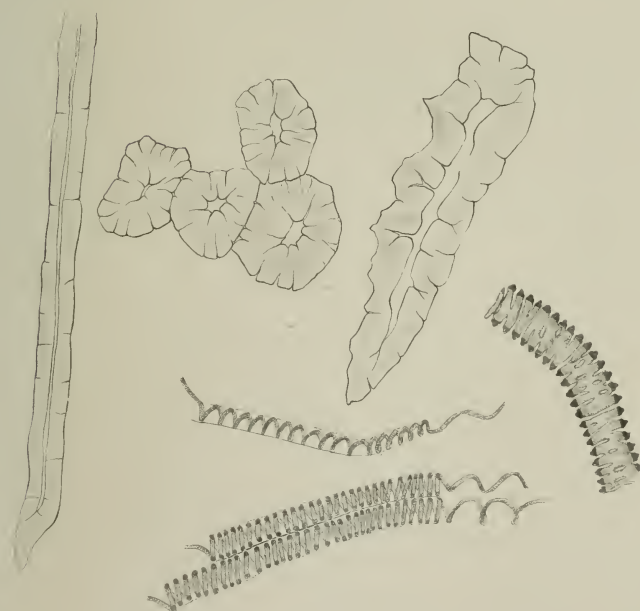


FIG. 2.—ELEMENTS OF FIBROVASCULAR BUNDLES OF PEAR. X 425.

DRAWN BY BURTON J. HOWARD.

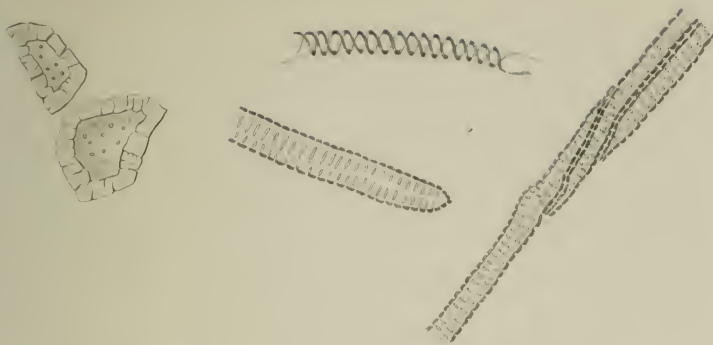


FIG. 1.—ELEMENTS OF FIBROVASCULAR BUNDLES OF QUINCE. X 425.

DRAWN BY BURTON J, HOWARD.

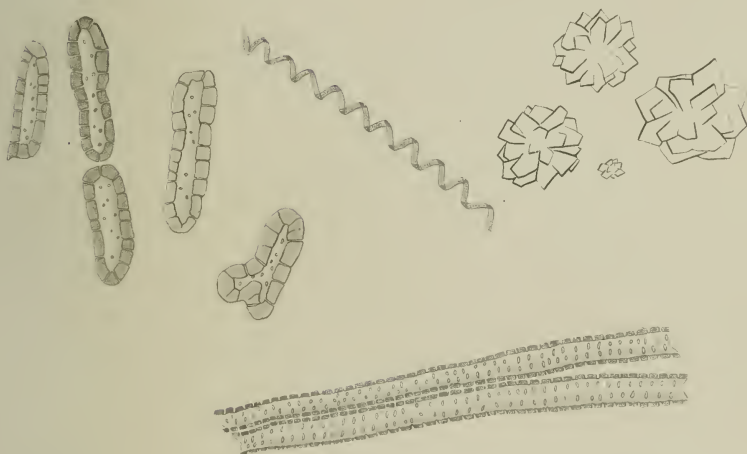


FIG. 2.—ELEMENTS OF FIBROVASCULAR BUNDLES OF PLUM. X 425.

DRAWN BY BURTON J, HOWARD.

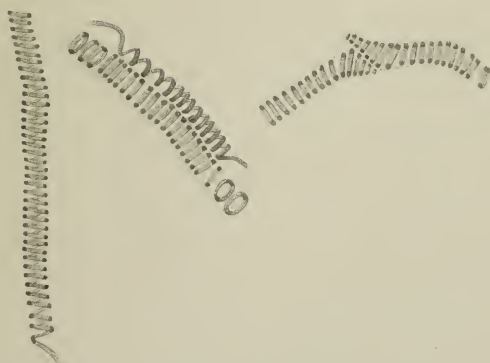


FIG. 3.—ELEMENTS OF FIBROVASCULAR BUNDLES OF GRAPE. X 425.

DRAWN BY BURTON J, HOWARD.

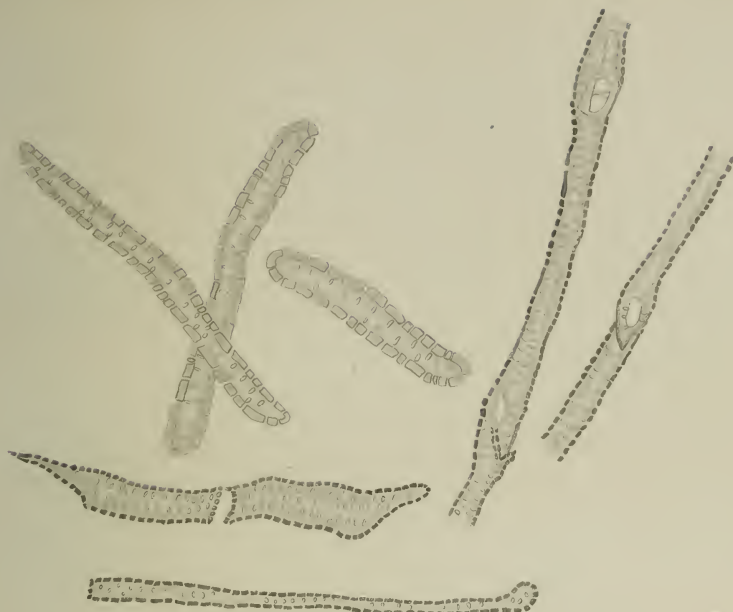


FIG. 1.—ELEMENTS OF FIBROVASCULAR BUNDLES OF PEACH. X 425.

DRAWN BY BURTON J. HOWARD.

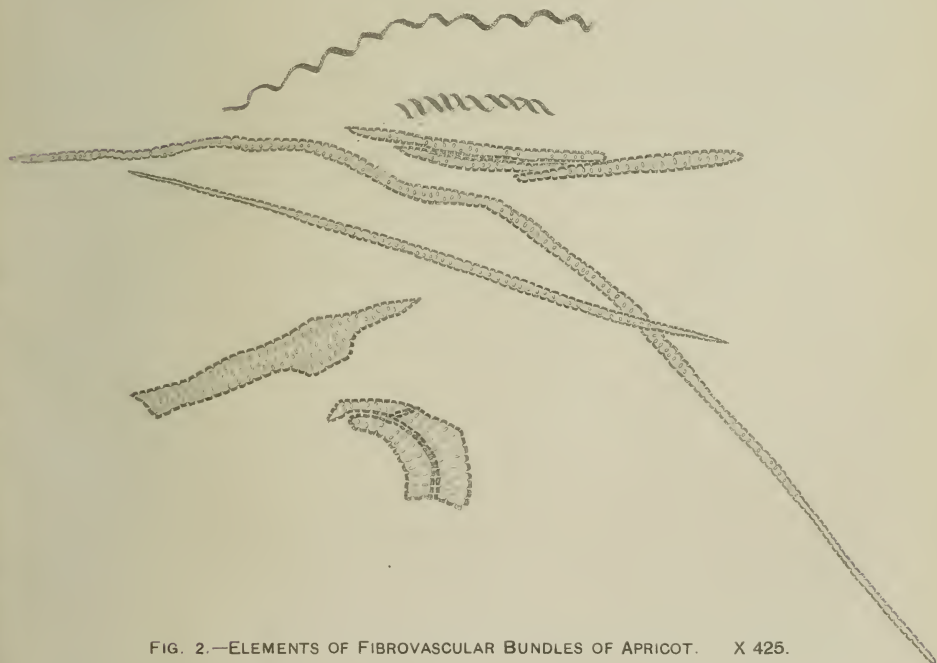


FIG. 2.—ELEMENTS OF FIBROVASCULAR BUNDLES OF APRICOT. X 425.

DRAWN BY BURTON J. HOWARD.

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John A. Myers

Born May 29, 1853; died April 8, 1901.

U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 67.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS

OF THE

EIGHTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.,

NOVEMBER 14, 15, AND 16, 1901.

EDITED BY

HARVEY W. WILEY,

SECRETARY OF THE ASSOCIATION.



WASHINGTON:

GOVERNMENT PRINTING OFFICE.

1902.

LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., January 31, 1902.

SIR: I have the honor to transmit to you herewith the manuscript of the proceedings of the eighteenth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 67, of the Bureau of Chemistry.

H. W. WILEY,
*Chief of the Bureau of Chemistry and Secretary of the
Association of Official Agricultural Chemists.*

HON. JAMES WILSON,
Secretary of Agriculture.

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Portrait of John A. Myers	Frontispiece
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PROCEEDINGS OF THE EIGHTEENTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

FIRST DAY.

THURSDAY—MORNING SESSION.

The eighteenth annual convention of the Association of Official Agricultural Chemists was held in Washington, D. C., beginning November 14, 1901. The meeting was called to order by the president, Mr. L. L. Van Slyke, in the lecture hall of the Columbian University.

MEMBERS AND VISITORS PRESENT.

Albert, A. D., Evening Star, Washington, D. C.
Allen, E. W., U. S. Department of Agriculture, Washington, D. C.
Allen, W. M., Assistant State Chemist, Raleigh, N. C.
Alsop, W. K., United States Leather Company, New York, N. Y.
Alwood, W. B., Agricultural Experiment Station, Blacksburg, Va.
Bache, A. W., U. S. Department of Agriculture, Washington, D. C.
Barnard, H. E., State Board of Health, Concord, N. H.
Beal, W. H., U. S. Department of Agriculture, Washington, D. C.
Benjamin, Marcus, The Smithsonian Institution, Washington, D. C.
Bigelow, W. D., U. S. Department of Agriculture, Washington, D. C.
Brown, B. E., U. S. Department of Agriculture, Washington, D. C.
Brown, E. W., U. S. Department of Agriculture, Washington, D. C.
Burd, J. S., U. S. Department of Agriculture, Washington, D. C.
Caldwell, G. C., Cornell University, Ithaca, N. Y.
Cameron, F. K., U. S. Department of Agriculture, Washington, D. C.
Carpenter, F. B., Virginia-Carolina Chemical Company, Richmond, Va.
Carr, Oma, Buena Vista Extract Company, Buena Vista, Va.
Church, C. G., Maryland Agricultural College, College Park, Md.
Cockrell, F. M., Jr., U. S. Department of Agriculture, Washington, D. C.
Cook, Wells W., U. S. Department of Agriculture, Washington, D. C.
Craighill, G. P., John H. Heald & Co., Lynchburg, Va.
Crampton, C. A., Chemist Internal Revenue, Washington, D. C.
Cullen, F. J., Photographer, Washington, D. C.
Davenport, E., University of Illinois, Champaign, Ill.
Davidson, R. J., Agricultural Experiment Station, Blacksburg, Va.
Donk, M. G., Assistant State Chemist, Tallahassee, Fla.
Doolittle, R. E., State Dairy and Food Department, Lansing, Mich.
Eaton, Edward N., State Analyst, Illinois Food Commission, Chicago, Ill.
Ewell, E. E., U. S. Department of Agriculture, Washington, D. C.
Ferris, E. B., Agricultural College, Mississippi.

- Fraps, G. S., Agricultural Experiment Station, Raleigh, N. C.
 Frear, William, Agricultural Experiment Station, State College, Pa.
 Fuller, F. D., Agricultural Experiment Station, Geneva, N. Y.
 Gardner, F. D., U. S. Department of Agriculture, Washington, D. C.
 Gascoyne, W. J., Baltimore, Md.
 Given, Arthur, U. S. Department of Agriculture, Washington, D. C.
 Gough, T. R., Maryland Agricultural College, College Park, Md.
 Guydon, T. P., Columbian University, Washington, D. C.
 Haley, E. J., A. Klipstein & Co., New York, N. Y.
 Hamilton, John, Department of Agriculture, Harrisburg, Pa.
 Haywood, J. K., U. S. Department of Agriculture, Washington, D. C.
 Herff, B. von, German Kali Company, New York, N. Y.
 Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
 Hopkins, C. G., Agricultural Experiment Station, Champaign, Ill.
 Howard, B. J., U. S. Department of Agriculture, Washington, D. C.
 Hurst, Lewis A., U. S. Department of Agriculture, Washington, D. C.
 Hurty, J. N., State Chemist, Indianapolis, Ind.
 Huston, H. A., State Chemist, Lafayette, Ind.
 Jenkins, E. H., Agricultural Experiment Station, New Haven, Conn.
 Jesse, R. H., State University, Columbia, Mo.
 Jordan, W. H., Agricultural Experiment Station, Geneva, N. Y.
 Kerr, G. A., Acme Bark Extract Company, Damascus, Va.
 Kilgore, B. W., State Chemist, Raleigh, N. C.
 King, A. F. A., Columbian Medical College, Washington, D. C.
 Krug, W. H., U. S. Department of Agriculture, Washington, D. C.
 Langley, C., Huyler's, New York, N. Y.
 Langworthy, C. F., U. S. Department of Agriculture, Washington, D. C.
 Law, L. M., Columbian University, Washington, D. C.
 Lawson, H. W., U. S. Department of Agriculture, Washington, D. C.
 Leach, A. E., State Board of Health, Boston, Mass.
 McCarthy, E. R., U. S. Department of Agriculture, Washington, D. C.
 McDonnell, H. B., State Chemist, College Park, Md.
 McGill, A., Laboratory Internal Revenue, Ottawa, Canada.
 McMurtrie, William, Royal Baking Powder Company, New York, N. Y.
 Machenheimer, G. L., Photographer, Washington, D. C.
 Magruder, E. W., Department of Agriculture, Richmond, Va.
 May, D. W., U. S. Department of Agriculture, Washington, D. C.
 Meng, James S., German Kali Works, New York, N. Y.
 Mitchell, A. S., Wisconsin Dairy and Food Commission, Madison, Wis.
 Moore, C. C., U. S. Department of Agriculture, Washington, D. C.
 Moore, J. S., Agricultural Experiment Station, Starkville, Miss.
 Morrison, W. G., F. S. Roysta Guano Company, Norfolk, Va.
 Munson, L. S., U. S. Department of Agriculture, Washington, D. C.
 Myers, W. S., Permanent Nitrate Committee for the United States and Colonies,
 New York, N. Y.
 North, J., Eimer & Amend, New York, N. Y.
 Norton, J. H., U. S. Department of Agriculture, Washington, D. C.
 Page, L. W., U. S. Department of Agriculture, Washington, D. C.
 Patrick, G. E., U. S. Department of Agriculture, Washington, D. C.
 Patterson, H. J., Agricultural Experiment Station, College Park, Md.
 Penny, C. L., Agricultural Experiment Station, Newark, Del.
 Price, T. M., Agricultural Experiment Station, College Park, Md.
 Reed, H. C., Stamford Manufacturing Company, Stamford, Conn.
 Reed, James, State College, Bozeman, Mont.

Robb, J. B., Maryland Agricultural College, College Park, Md.
 Robertson, A., Laidlaw, Mackill & Co., Richmond, Va.
 Ross, B. B., State Chemist, Auburn, Ala.
 Runyan, E. G., U. S. Department of Agriculture, Washington, D. C.
 Scovell, M. A., Agricultural Experiment Station, Lexington, Ky.
 Seidell, A., U. S. Department of Agriculture, Washington, D. C.
 Sherman, F., Department of Agriculture, Raleigh, N. C.
 Spencer, G. L., U. S. Department of Agriculture, Washington, D. C.
 Stedman, J. M., State University, Columbia, Mo.
 Straughn, M. N., Maryland Agricultural College, College Park, Md.
 Street, J. P., Agricultural Experiment Station, New Brunswick, N. J.
 Stuart, Duncan, U. S. Department of Agriculture, Washington, D. C.
 Sweetser, W. S., Hampton Institute, Hampton, Va.
 Terne, Bruno, Union Abattoir Company, Baltimore, Md.
 Tibbitts, J. H., Washington, D. C.
 Tolman, L. M., U. S. Department of Agriculture, Washington, D. C.
 Trescot, T. C., U. S. Department of Agriculture, Washington, D. C.
 Van Dyke, J. H., U. S. Department of Agriculture, Washington, D. C.
 Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
 Veitch, F. P., U. S. Department of Agriculture, Washington, D. C.
 Voorhees, E. B., Agricultural Experiment Station, New Brunswick, N. J.
 Watts, Henry C., Marsden Company, Philadelphia, Pa.
 Wheeler, H. J., Agricultural Experiment Station, Kingston, R. I.
 White, H. C., Agricultural Experiment Station, Athens, Ga.
 White, H. F., University of Vermont, Burlington, Vt.
 Wiley, H. W., U. S. Department of Agriculture, Washington, D. C.
 Wiley, S. W., Hatch Experiment Station, Amherst, Mass.
 Willard, J. T., Manhattan, Kans.
 Williams, C. B., Department of Agriculture, Raleigh, N. C.
 Williams, Walter, Columbia, Mo.
 Winton, A. L., Agricultural Experiment Station, New Haven, Conn.
 Withers, W. A., Agricultural Experiment Station, Raleigh, N. C.
 Woodman, A. G., Institute of Technology, Boston, Mass.
 Woods, C. D., Agricultural Experiment Station, Orono, Me.
 Yocum, Frank H., Yocum Manufacturing Company, Newark, N. J.

ORDER OF BUSINESS.

The following order of business was observed:

The president's address.

Reports of the referees in the following order:

First. Report on nitrogen.

Second. Report on potash.

Third. Report on phosphoric acid.

Fourth. Report on soils.

Fifth. Report on ash.

Sixth. Report on foods and feeding stuffs.

Seventh. Report on liquor and food adulteration.

Eighth. Report on dairy products.

Ninth. Report on sugar.

Tenth. Report on tannin.

Eleventh. Report on insecticides.

Twelfth. Reports of special committees (abstract committee, food standards, fertilizer legislation, volumetric standards).

Mr. Van Slyke addressed the convention as follows:

PRESIDENT'S ADDRESS.

GENTLEMEN OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS: This is the eighteenth annual convention of our association. To one making a review of the proceedings of the association for these years one striking fact becomes prominent, namely:

THE EXPANSION OF THE WORK.

The original scope of the work of the association was limited to a study of methods relating to the determination of nitrogen, phosphoric acid, and potash in commercial fertilizers. Fifteen years ago a process of evolution began and has continued until to-day we have the original 3 divisions increased to 11. But the actual growth of work is greater than that implied by this statement, since each of the added divisions has involved a group of determinations rather than a single one. For example, the single subject of dairy products is subdivided into three parts—butter, milk, and cheese—while each of these involves several distinct determinations. The subject of liquor and food adulteration has been split up into 15 separate sections. Originally 3 committees took charge of all the work of the association; to-day there are 11 referees and 25 associate referees, not to mention standing committees on food standards, volumetric standards, and fertilizer legislation, which are carrying on additional work.

The primary impulse in the direction of expansion was given by Dr. Wiley in his address as president of the association in 1886, when he said:

A first step forward has certainly been taken, but the road before us is still a long one. * * * The good results of our meetings might be widely extended if our investigations were made to include a wider range of subjects. Every problem connected with chemical agricultural analysis falling within the range of the studies of anyone connected officially with the agricultural interests of the country seems to me to be a proper theme for our discussion here. Thus, all problems relating to the adulteration of food, the general method of agricultural analysis, and all other matters which concern in common the analyst and the public could be included among the duties and privileges of this body.

This suggestion was at once acted upon and the development of our work followed. We can repeat now with perhaps more emphasis the statement made fifteen years ago: "The road before us is still a long one."

CHANGES IN CHARACTER OF WORK.

During these years there has gradually come a marked change in the character of the chemical work done by the association apart from that involved in its expansion. At first the work was comparatively simple in kind, involving a minimum of research work by individuals and that not especially difficult; but with advance in methods the easier ground has been covered and there is arising a demand for more original investigation, increasingly difficult and complicated in character. Some of our methods of analysis must remain unimproved, unless individual members of the association devote much time to special research. More and more new methods and modifications must be worked out in our individual laboratories and then presented to the association for final testing. To make my meaning more precise, let me illustrate, using the analysis of cheese as an example. The association methods as given at present provide for no determination of the various classes of nitrogen compounds. We know that what we were formerly pleased to call casein in cheese is really a complex mixture made up of many different nitrogen compounds, such as paracasein, nuclein, albumoses, peptones, amides, ammonia, and perhaps others. Something must be done toward isolating and identifying the individual compounds before we can hope to provide effective methods for their estimation, and this involves an immense amount of hard work, which must be done largely by individual initiative. Such work the association can not easily lay out and assign, but until such work is accomplished there is little hope for advancement in the development of methods.

I believe it should be the policy of this association to encourage even more than it has in the past the presentation of papers embodying results of research in all lines bearing in the least upon the study of methods. Sooner or later our work in the perfection of analytical methods will depend entirely upon advanced work of this character. Each year makes this fact more prominent.

Incidentally, in connection with such changes in the character of our work, it may be observed that the agricultural chemist of the future who would be most useful needs a much broader preparation for work than that of a mere analyst. A dozen years ago the demand for agricultural chemists was so great in proportion to the supply that an efficient routine analyst with some experience along agricultural lines was at a premium. Good routine analysts are plentiful now, but there is a growing demand for men who have had a broad and thorough training and possess also the proper temperament for research work. The chemical problems that come to us are constantly growing more complicated and difficult. We are being more and more irresistibly drawn into the fascinating but trying field of life chemistry.

A dozen years ago or less scarcely an experiment station chemist would have dared to devote his time to a problem whose results could not be quickly interpreted in dollars and cents to the farmers. But we are gradually getting a little away from such conditions, and some now dare to approach the solution of questions that partake more of the character of advanced science.

FURTHER EXTENSION OF WORK.

The time may not yet be ripe for further expansion in the scope of our work, but it may not be out of place to make a suggestion at this time, looking forward to a desirable extension as soon in the future as may be found practicable. The suggestion I have to make is amply covered by that clause of our constitution which states that one object of the association is "to afford opportunity for the discussion of matters of interest to agricultural chemists." I believe that the time is near at hand when the association should make some effort to encourage and provide for the presentation and discussion at our meetings of papers pertaining to lines of investigations, largely chemical, but not relating necessarily to methods of analysis. Many of our members are carrying on valuable experimental research work in studying chemical problems relating to various lines of agricultural work. Several stations have in hand investigations bearing on some phase of the chemistry of animal nutrition. One station has done much work of interest in studying the relation of line compounds to plant nutrition, and another has been investigating the influence of various forms of plant food upon the chemical composition of fruits and plants. At least two of our members have done work relating to the manufacture and composition of cider and cider vinegar. Two stations are studying the biochemical problems of cheese ripening, and I might continue for some time naming similar lines of research, but these suffice for illustration. Now, there is really no other place or occasion where these subjects can be so properly presented and discussed as at the meetings of the Association of Official Agricultural Chemists, because only here can they receive thoroughly intelligent and appreciative attention and consideration in the presence of a large body made up of agricultural chemists. Neither in the chemical section of the American Association for the Advancement of Science, the meetings of the American Chemical Society, those of the Society for the Promotion of Agricultural Science, nor in the Association of Agricultural Colleges and Experiment Stations, can those subjects of most interest to us as chemists receive the amount or kind of attention that can be given to them here. I believe some time each year could be devoted to such additions to our progress without unduly prolonging our meetings or taking time from the features that constitute the bulk of our work.

EXPEDITION IN PROCEEDINGS OF CONVENTION.

Permit me to suggest that it is possible for us to expedite our business and economize time without in any degree slighting our work. Undoubtedly we have all noticed opportunities for improvement in this direction during the past. I desire to call attention to a few points in this connection.

First. In too many instances the reports of referees have been presented in forms so incompletely digested as to consume time needlessly. The ideal report of a referee should exclude every detail that is not helpful in enabling members to form an intelligent opinion of the matter presented. Many details may go into the printed report that need not be read on the floor. What we want presented on the floor of our convention are sharp, clear, comprehensive statements of results, with only such detailed explanations as are needed to make those statements readily understood. Too often in the past time has been unprofitably consumed by the referee in reading every figure of every individual worker, even when printed tables containing all the data were in our hands. There has been great improvement in this respect during the past ten years, but a still greater improvement is in order.

Second. If time may not be saved, interest at least will be increased if referees and all speakers will develop enough lung power to let others know what they are saying.

Third. In our discussions it too often happens that old ground which is or ought to be familiar is repeatedly gone over year after year by some speakers, usually by those who have not attended the meetings regularly. It may be safely assumed that the general facts of elementary chemistry do not need repetition here. It would yield beneficial results if all of us would take time now and then to look over the proceedings of previous meetings.

Fourth. Extreme care should be taken in the selection of the chairman of the committee on recommendations of referees. The duties of the place are arduous at best and especially in presenting matters to the convention, which requires a strong voice, system, ability to state facts sharply and clearly, self-possession, an understanding of the facts presented, patience, alertness, and omnipresence. The wrong man in this place can easily waste one or two hours of the convention's time. With increase of work it might be wise to divide this labor among three committees. A plan will probably be brought forward on this and associated points.

Fifth. One other suggestion in connection with saving time is the matter of promptness in attendance at the opening of every session.

Of all the associations with which I have had any experience, this is unquestionably the hardest working. The foregoing suggestions

have been made not with the intention of making us work still harder, but in the hope of so conserving our time and efforts that our results may be no less effective with the expenditure of less time and labor.

DEATH OF JOHN A. MYERS.

During the past year there has passed from life one who was from the beginning intimately connected with the work of this association, Mr. John A. Myers, president in 1889. It is highly fitting that a special evening session should be held by us in honor of his memory.

DISCONTINUANCE OF THE COMMITTEE ON ABSTRACTING.

The discontinuance of the committee on abstracting is suggested, as this work is now fully provided for in the Office of Experiment Stations.

CHEMISTS AS PRACTICAL MEN.

In conclusion, I want briefly to consider one more topic, the discussion of which in this place may seem a departure from our traditions, but its justification will appear in due time. The topic is "Chemists as practical men."

I remember of once hearing, at a meeting which I attended, the statement that chemists in general were quite outside the circle of those possessing plain, common, everyday horse sense. It was emphatically stated, with an effort at dramatic eloquence, that the training and habits of life of the chemist led him to such retirement from contact with the world and isolation from his fellow men that he was necessarily and unavoidably deficient as a man of affairs, sadly lacking in executive ability and knowledge of men, etc.—in fact he was pictured as a kind of intellectual babe in the wood when he went outside his laboratory. This was an echo of the old traditional cry that a man of scientific training could not possibly be a practical man in the affairs of everyday life. The conclusion drawn by the speakers was that the chemists could not be expected and should not be allowed to occupy positions calling for executive ability and acquaintance with the ways of men, places requiring the exercise of good judgment and plain common sense. It was also concluded that chemists, having no sense outside the laboratory except that of smell, should not obtrude their impracticable and valueless opinions in public councils. They might be seen, but not heard. Under the circumstances, I thought it best to keep still. It has seemed proper, therefore, that a defense should be entered where the audience would be more fully sympathetic.

I can not too strongly urge you who have not done so to read an article entitled "The man of science in practical affairs," by Dr. F. W.

Clarke, in *Popular Science*, February, 1900. He discusses the question in a most thorough and convincing manner.

In closing, it may be pertinent to state that more than one-third of the directors of our experiment stations and a few college presidents have been active members of this association. The fact is that if a man has in him the elements of being practical, a chemical training seems peculiarly adapted to develop that side of his personality.

As Dr. Clarke says, in the article referred to:

At bottom the scientific training is a training in clear, thorough, precise statement, accurate observation, the verification of evidences, and the ascertainment of truth. Why should its recipient be unfitted for practical things? Good administration, the effective transaction of business, implies system, exactness, the judgment of evidence upon its merits, and the prompt solution of problems as they arise, and to each of these requisites the scientific education is directly related. What other training is less likely to produce dreamers or more likely to develop efficient men?

APPOINTMENT OF COMMITTEES.

The PRESIDENT. It has been the custom at this point of the proceedings to appoint committees, and if there is no objection I will announce these appointments:

The committee to wait upon the Secretary and Assistant Secretary of Agriculture, to inform them that we are in session, and that the members of the association will be pleased to have them meet with us, will consist of Messrs. Kilgore and Scovell.

Committee on resolutions: Messrs. Frear, Wheeler, and Jenkins.

Committee on nominations: Messrs. Huston, Woods, and McDonnell.

Committee on amendments to the constitution: Messrs. Wheeler, Caldwell, and Winton.

Mr. WILLIAMS. For some years past the association in its preliminary actions has followed the practice of securing careful consideration and expression—the need of which had been previously demonstrated—in addition to that given by the referee, in regard to the appointment of a committee on recommendations. Within the last two or three years our work has branched out in such a manner as to involve the consideration of a much greater number of different classes and kinds of methods than this association has heretofore undertaken to examine, and it appears that a number of the members of the association think that to expect one committee on recommendations to give mature consideration to the recommendations on the large variety of subjects treated is not just to the committee nor to the subjects. I would therefore move, Mr. President, that a committee of three be appointed by the Chair to consider and report to the association at this morning's

session how many committees should be appointed to act on the recommendations of referees upon the several classes of subjects.

The PRESIDENT. The motion has been made and seconded that a committee be appointed to report on increasing the number of committees on the recommendations of referees. The motion is carried. I will appoint Messrs. Frear, Wheeler, and Kilgore.

The next item in the regular order of business is the presentation of the report on nitrogen.

Mr. WHEELER. If there is no objection, Mr. President, I should like to introduce a motion.

The PRESIDENT. If there are no objections you may have the floor for that purpose.

Mr. WHEELER. In view of the fact that the number of referees has been increased perhaps two or three times, and the difficulty that any one man has in selecting these referees, I desire to offer an amendment to the constitution. The proposition has been made that the appointment of referees be referred to a select committee rather than to the president of the association. The proposed amendment is as follows:

Resolved, That section 4 of the constitution be amended by substituting for the word "president," in the first line of the section, the words "executive committee."

I move that a committee be appointed to consider this resolution.

Mr. KILGORE. I would call for the reading of that part of the constitution which it is proposed to change.

The PRESIDENT. The member will please read the section as it now is and the correct change which he proposes.

Mr. WHEELER (reading):

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

According to the proposed change it would read:

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

The PRESIDENT. Has this motion been seconded?

Mr. FREAR. I second the motion.

Mr. KILGORE. What article of the constitution gives authority for amending it?

The PRESIDENT. Section 10.

All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

Are there any remarks upon the motion in regard to the appointment of a committee to which shall be referred the resolution to amend section 4 of the constitution by placing the appointment of referees in

the hands of the executive committee? If not, all who are in favor will say "Aye." Opposed, the same. It is carried.

Mr. FREAR. I would like to introduce another resolution affecting the constitution, and move that it be referred to the consideration of the same committee. This resolution affects section 7, which at present reads as follows:

(7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

The association has of late years taken up quite a large amount of work affecting the official investigation of the various kinds of trial inspection. It seems to me that the same thing can be said in regard to the soil-inspection work, in which a large number of us are concerned. I would therefore propose, Mr. President, the following:

Resolved, That section 7 of the constitution be amended by substituting, in the first line, for the words "fertilizer analyses," the words "analysis used in official inspection," and in the third line for the words "fertilizer work" the words "the particular inspection affected."

The PRESIDENT. A motion has been made to change the constitution in the manner specified. Are there any remarks?

Mr. EWELL. I would like to suggest that some time limit be inserted. Should it not be a year, or a certain length of time?

The PRESIDENT. That would probably be considered by the committee.

Mr. WINTON. Is there anything in the proposed change that would prevent the introduction of additional propositions?

Mr. FREAR. Not at all.

The PRESIDENT. This will be referred to the committee on amendments to the constitution, Messrs. Wheeler, Caldwell, and Winton. Mr. Perkins has said that he would have his report here, but it has not yet been received, and the next in order is the report of the referee on potash. Is he present?

Mr. ROSS. The referee handed the report to me for presentation to the association and asked me to hand it to the secretary, who requests that I should read the report.

Mr. EATON. I wish to present to the association a letter from Mr. J. B. Noble, as follows:

STATE OF CONNECTICUT, DAIRY COMMISSIONER'S OFFICE,

Hartford, November 8, 1901.

DEAR SIR: At a meeting of the National Association of State Dairy and Food Commissioners held in Buffalo October 15, 16, and 17, 1901, the following persons were appointed as a committee to represent the association before the National Association of Agricultural Chemists, to be held in Washington: Dr. E. N. Eaton, of Illinois; Dr. R. M. Doolittle, of Michigan; Dr. A. S. Mitchell, of Wisconsin; Dr. William Frear, of Pennsylvania.

Yours, respectfully,

J. B. NOBLE, *Secretary.*

Dr. E. N. EATON, *Chicago, Ill.*

The PRESIDENT. We have this letter from Mr. Noble. What action shall be taken on it?

Mr. FREAR. I was made a member of this committee, but have just received my notification and am not prepared to submit a report. I was not in attendance with the committee during the national convention in Buffalo at the time its action was taken, but since I am chairman at present of the committee of this association charged with the consideration of food standards, I would offer this resolution for the consideration of the association:

Resolved, That the Association of Official Agricultural Chemists welcomes the action of the National Association of State Dairy and Food Commissioners in appointing a committee of its members to present to our association its views upon the subject of food standards—the formulation of which has already been undertaken by this association for the guidance of its members—and upon the methods of detection of food adulteration.

The PRESIDENT. You have heard the resolution which has been presented to the association with reference to this matter. What action shall be taken with regard to it?

Mr. EATON. I see that the resolution covers, at least in part, the ground that our association has already gone over in the establishing of standards, and it would at least be open to criticism as to the point of practice to publish the different reports. I do not believe that the question of standards was the only one my associates had in mind. The question of the adoption of food analyses was also brought out. I am considerably interested in this topic, yet I shall be as brief concerning it as possible and take up no more time than necessary. I think that possibly this resolution might be adopted to cover the question of the methods of analyses as well as standards for food products and I am sure the members of the committee will be glad to cooperate with the committee appointed by this association to look after the methods of analyses and standards of food products and concur with them or give any advice their experience may have made them competent to give.

Mr. FREAR. I shall be glad to so change the wording of the resolution in the manner suggested by Mr. Eaton, and to have it cover the subject of food sanitation and also the subject of the methods of analyses for the detection of adulteration.

The PRESIDENT. The motion is upon the adoption of this resolution.

Mr. KILGORE. I don't know that I quite understand the import of this resolution. This association is working especially upon the subject of methods. It has been on that question for some time, and last year enlarged the number of men engaged on it to 15, and this year we have given a large part of our time to that work and there will be presented to this association at least the methods advised. Do I understand that the committee from this association is going to confer

with the committee of the other societies with reference to these particular methods?

Mr. FREAR. In regard to the resolution I might explain further that my purpose in introducing it was to formally welcome an attitude of cooperation with the movement which has been started through the officials organized under the title of National Association of State Dairy and Food Commissioners, who are in charge of the examination of State laws controlling the various human foods in the several States, but not to indicate the method specifically by which this cooperation should be attained. I hardly thought that such a statement was necessary in the resolution for the reason that every chemist who is appointed by this National Association of State Dairy and Food Commissioners is entitled to membership in this body, and therefore is part of this working organization, and as such would be entitled to present any views to any committee of this body just as any other member is entitled, and, also, coming in a representative capacity, he would be entitled to hearing and representation before final action is taken, and any words uttered by him would be given due weight.

Mr. WHEELER. Is there not a committee on resolutions? If so, I would suggest this matter be referred to that committee.

The PRESIDENT. The matter will be referred to the committee without motion, if there is no objection.

We will now take up the report on potash, prepared by Mr. C. L. Hare.

REPORT ON POTASH.

By C. L. HARE, *Referee*.

In the absence of recommendations from the association the referee planned the work on potash for 1901 so as to include the following investigations:

(1) Continuation of the previous years' inquiries, suggested by Mr. F. B. Carpenter, concerning the probable failure of the Lindo-Gladding method to obtain from mixtures of acid phosphate and potash salts the theoretical amount of potash added.

(2) The use of ammonium chlorid as a possible aid in securing solution of all potash present in such mixtures.

(3) The referee for the year 1899 described briefly a method for the determination of potash based upon the use of barium carbonate for precipitation from neutral solution. Later, he abandoned that method and turned his attention to one suggested by that, i. e., the use of milk of lime as the precipitant. His method has given such excellent results in the laboratory of the Alabama station that the present referee felt justified in offering it to the association for trial. Consequently the method, as described below, was included in the work.

During the spring over thirty circular letters were sent out to chemists who, it was thought, might be interested. Twelve analysts expressed a willingness to cooperate. The following letter of instructions was sent out with each set of samples:

DEAR SIR:

I have mailed you to-day two samples for official potash work.

Sample No. 1 is chemically pure potassium chlorid. Sample No. 2 is a mixture of chemically pure potassium chlorid and acid phosphate in the proportion of 5 to 95.

1. Determine potash in No. 1 by the official method.

2. (a) Determine water-soluble potash in No. 2 by the official method.

(b) Determine water-soluble potash in No. 2 by the official method with the exception that 5 to 10 grams of ammonium chlorid should be added to the weighed sample before making solution.

(c) Determine water-soluble potash in No. 2 by the following method: Make the solution according to the official method, and while the solution is still hot add milk of lime until alkaline. Boil for a few minutes, cool, make up to the mark, measure out an aliquot portion, acidify with hydrochloric acid, and evaporate with platinum solution; wash with alcohol and Gladding "wash" as in official method.

3. Make blank determinations on reagents, using amounts required for regular determinations.

In reporting results please state what filter was used for filtering potassium platonic chlorid. If convenient use the Gooch filter.

State also the method by which the asbestos was prepared for use. Report amount of insoluble residue after washing out K_2PtCl_6 with hot water.

Please report all observations that appear to be of interest.

C. L. HARE, *Referee*.

Sample No. 1 was potassium chlorid. Sample No. 2 was prepared by mixing intimately acid phosphate and chemically pure potassium chlorid in the proportion of 95 acid phosphate to 5 potassium chlorid. Reports were received from eight laboratories, representing the work of twelve analysts. The following table shows in detail the results obtained:

TABLE 1.—*Results of analysis of potassium chlorid and of the mixture of potassium chlorid and acid phosphate.*

Analyst.	Sample No. 1, C. P. KCl.	Sample No. 2, 95 acid phosphate and 5 KCl.		
		Lindo-Gladding method.	Milk-of-lime method.	Lindo-Gladding with NH_4Cl .
J. Q. Burton, Georgia.....	62.82	3.10	3.29	3.13
		3.03	3.39	3.11
	62.82	3.07	3.34	3.12
R. G. Williams, Georgia.....			3.19	
			3.11	
			3.18	
			3.16	
I. D. Sessums, Mississippi.....	62.60	3.15	3.18	3.39
	62.60	3.14	3.14	3.35
	63.10	3.20	3.19	3.34
	62.80	3.14	3.18	3.37
		3.14	3.13	3.33
		3.15	3.14	3.34
		3.14	3.16	3.34
		3.12	3.19	3.32
O. W. Knight, Maine.....	62.80	3.15	3.15	3.35
	62.96	3.14	3.13	3.18
	62.96	3.13	3.12	3.09
	63.00			
C. D. Holley, Maine.....	62.97	3.14	3.13	3.14
	63.00	3.14	3.13	3.24
	63.00	3.16	3.155	3.21
C. H. Jones, Vermont.....	63.00	3.15	3.14	3.23
	63.21	3.23	3.11	3.28

TABLE 1.—*Results of analysis of potassium chlorid and of the mixture of potassium chlorid and acid phosphate—Continued.*

Analyst.	Sample No. 1, C. P. KCl.	Sample No. 2, 95 acid phosphate and 5 KCl.		
		Lindo-Gladding method.	Milk-of-lime method.	Lindo-Gladding with NH_4Cl .
C. I. Boyden, Vermont		3.20	3.06	3.29
		3.04	3.12	3.24
		3.12	3.09	3.27
F. B. Carpenter, Virginia.....	62.90	3.10	3.05	3.14
E. L. Parker, Virginia	62.92	3.08	3.08	3.12
T. R. Gough, Maryland.....	63.80	3.08	3.17	3.22
	62.20	3.03	3.15	3.24
	63.00			
	63.00	3.05	3.16	3.23
R. F. Hare, New Mexico		3.09	3.07	
		3.07	3.08	
		3.08	3.06	
		3.11	3.04	
C. L. Hare, Alabama.....		3.09	3.06	3.06
	62.91	3.19	3.24	3.17
		3.16	3.24	3.15
		3.14	3.25	3.14
		3.18	3.26	3.13
		3.09	3.28	3.13
		3.18	3.29	3.09
Means	62.91	3.16	3.26	3.14
	62.95	3.12	3.15	3.19

COMMENTS OF REFEREE.

Careful analyses made in the laboratory of the Alabama station of the acid phosphate used in making the mixture from which Sample No. 2 was drawn showed 0.2 per cent potash. Based on the analysis of the acid phosphate and of the potassium chlorid, the theoretical amount of potash in No. 2 was 3.35 per cent.

On reference to the table it is seen that the official method fails to remove the theoretical amount of potash—the difference between theory and the average of results being 0.23 per cent. Results by this method are within 0.18 per cent of each other, and with such close agreement no satisfactory explanation is at hand as to why results are so far below theory.

While the addition of ammonium chlorid before making solution gives an increase of 0.07 per cent potash, the results reported do not warrant the conclusion that ammonium chlorid may be used with advantage. One disadvantage is that the large amount of ammonia salts left after evaporating the solution with sulphuric acid may cause loss from sputtering.

The referee would direct especial attention to the results obtained by the milk-of-lime method. It will be observed that these results, excluding one determination, agree within 0.21 per cent of each other. The average of results agrees within 0.02 per cent of the average of results obtained by the official method. This speaks highly for a method which is having its first trial in the hands of the majority of the analysts.

While the direct evaporation of the solution with platinum solution leaves a rather large amount of lime salts, none of the analysts have reported trouble in the final washings. In washing this residue, analysts who have little experience with the method are apt to subject it to too vigorous rubbing. Experience teaches that excessive pulverization is unnecessary, and will possibly result in loss of potash, caused by the finely divided potassium platinic chlorid passing through the filter. It is the opinion of the referee that in hands familiar with it the method will remove a slightly higher per cent of potash from mixtures of acid phosphate and potash salts than will the Lindo-Gladding method.

The small amount of manipulation required in the method, the fact that evaporation with sulphuric acid—a fruitful source of error—is avoided, the rapidity of the method, and finally the excellent results obtainable, recommend the method as one which may be brought to a very high degree of usefulness. It seems to be applicable to the class of goods on which it was tried, and experiments conducted at the Alabama station indicate that it can be used as successfully on fertilizers containing organic materials of various kinds.

The following table includes results obtained by the writer by applying the method to fertilizers containing such organic materials as cotton-seed meal, tankage, blood, bones, and tobacco stems. The potash content of the samples was, with one exception, unknown, and in parallel columns is given the per cent of potash by the Lindo-Gladding method.

TABLE 2.—*Results on fertilizers containing organic materials.*

Sample.	Lindo-Gladding method.	Milk-of-lime method.
1.....	2.55	2.68
2.....	1.80	1.80
3.....	1.97	2.33
4.....	1.70	1.74
5.....	1.35	1.34
6.....	1.94	1.80
7.....	1.67	1.67
8.....	2.36	2.38
9.....	1.49	1.42
10.....	1.48	1.42
11.....	1.33	1.38
12 (official potash sample for 1900)	5.40	} 5.42 5.44 5.45

Following is the method employed:

Ignite 10 grams of sample with sulphuric acid, and after cooling add 4 or 5 cc sulphuric acid (1-1) and gently warm the dish. The sample may now be easily removed from the dish, and the small amount of free sulphuric acid interferes in no way with the following stages of the process.

Boil the ignited sample with about 350 cc of water for thirty minutes, and while hot add milk of lime till alkaline. Precipitation of all the interfering salts is complete. Cool, make up to 500 cc, take an aliquot portion, acidify with hydrochloric acid, and evaporate direct with platinum solution. Wash with alcohol and ammonium-chlorid wash water, as in the Lindo-Gladding method. Little more time is required for the washing than in the Lindo-Gladding method.

It was found that rubbing the residue quite vigorously with a rubber pestle caused low results, presumably from the passing of finely divided potassium platinic chlorid through the filter.

If a somewhat larger amount of ammonium-chlorid wash water be used than in the Lindo-Gladding process, gentle rubbing with the finger or with a rubber-tipped glass rod suffices to divide the residue finely enough for the wash water to completely dissolve the salts of lime present.

RECOMMENDATIONS.

The referee would suggest:

- (1) Investigations into the causes of the low results obtained by the official method.
- (2) That the "milk-of-lime" method be submitted for investigation the ensuing year.

Mr. Ross. With regard to the "milk-of-lime" method, to which considerable attention has been given by the referee, I would say that this process suggested itself to me shortly after the preparation of my report on potash in 1899, and immediately a series of tests was instituted in the Alabama State laboratory to determine the practicability of this modification. This process is, of course, only applicable to mixtures of acid phosphate and potash salts, or to fertilizers containing organic materials, in which case saturation of the material with sulphuric acid and incineration are resorted to before effecting the solution of the potash.

In the present official method for determining total potash in organic mixtures, the material is incinerated after treatment with sulphuric acid, and at a subsequent stage of the process evaporation and ignition must be again resorted to in order to remove the ammonia and ammonia salts which have been employed as precipitants. In the "milk-of-lime" process this second ignition is avoided, precipitation of phosphates, lime, alumina, etc., being effected by means of the milk of lime. Mr. Hare took this matter up even before his appointment as referee, and has performed a large amount of laborious, careful, and painstaking work in the testing of this method.

Mr. WILEY. I have a communication from Mr. Peters on this subject, which I took the liberty of opening, and I would like to submit this for publication. I do not think it is necessary to read it, although there are only two pages.

LEXINGTON, KY., November 9, 1901.

MY DEAR SIR: I have recently had occasion to try the determination of potassium oxid in a vegetable substance by the method of charring with sulphuric acid and burning, as described on page 22 (2 b), Bulletin 46, revised edition. The result was that less potash was obtained in this way than by boiling out with water in the manner prescribed for mixed fertilizers without previous burning. This recalls former experiences in the same line, which I think I have mentioned to you in a former letter, and makes me think that we might do well to drop the paragraph above cited from our methods, leaving determinations of total potash in such materials to be made by the methods described for the ashes of plants, and using only the method of boiling out with water for fertilizers. The old experiments referred to above were made upon tobacco stems and the results were published in the Eighth Annual Report of the Kentucky Station, 1895, page xxiii, and I have had the same experience since then. The following are the figures:

Tobacco stems, No. 2935—Air-dried sample.

	Per cent.
K ₂ O by boiling 10 grms. with 300 cc water one-half hour (Bull. 46, rev. ed., p. 21).....	9.32
K ₂ O by charring with H ₂ SO ₄ , treatment with HCl, etc. (Bull. 46, rev. ed., p. 22).....	9.08
K ₂ O by incineration and analysis of the ash.....	9.18

The trouble seems to me to be in not getting the mass fully dissolved by treating with hydrochloric acid and boiling one-half hour with 300cc of water. After incinerat-

ing with sulphuric acid the mass obtained is hard and lumpy and does not readily dissolve. I believe this is a weak point in our methods and should be looked after, though it is not of very great importance, since the method is seldom needed.

Very respectfully,

ALFRED M. PETER.

Mr. C. L. HARE,

*Referee on Potash, Association of Official Agricultural Chemists,
Care of Dr. H. W. Wiley, Washington, D. C.*

The PRESIDENT. The report is before you for discussion.

Mr. MAY. I would like to say that I am writing a report of this meeting for the Experiment Station Record, and will ask the referees to leave their papers with Dr. Wiley and not take them home, as some have done in the past.

The PRESIDENT. I hope the various referees will adhere to the request made. If there are no remarks, I will say that opportunity will be given for the discussion of any of the reports of the referees at the time recommendations are submitted by the committee. We will proceed next to the report on phosphoric acid.

Mr. WILEY. I have a report sent by Mr. Miller, of Florida, who is unable to be present. I am suffering a little with a cold and I would ask someone to read this.

The report was read by Mr. Runyan, as follows:

REPORT ON PHOSPHORIC ACID.

By H. K. MILLER, *Referee.*

An examination of the work done the past four years in connection with the estimation of phosphoric acid by this association shows that much has been accomplished. The volumetric method has received careful study and has now passed beyond the experimental stage, and seems to give more reliable results in the hands of those experienced in its use than the gravimetric method. In view of this the referee for this year decided it would be well to pay greater attention to some other phase of the phosphoric acid work. The modification of the volumetric method as proposed by the referee of last year yielded such good results that it was deemed well to incorporate it in the instructions for this year with little change.

The usual letter was sent out to those thought to be interested, requesting cooperation in the work on phosphoric acid for 1901. Some fifty replies were received, and among these fifteen expressed a willingness to assist in the work and requested a set of samples. Samples were prepared and sent out with the following letter of instructions:

INSTRUCTIONS FOR THE WORK ON PHOSPHORIC ACID FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1901.

Three samples have been prepared for the phosphoric acid work this year.

No. 1 is a basic slag.

No. 2 is ground phosphate.

No. 3 is a mixture of salts containing phosphoric acid, alumina, and iron.

All of No. 3 is to be placed in a liter flask, a little nitric acid added, and made up to mark with water. Fifty cubic centimeters of the solution will correspond to 0.5 gram of substance.

TOTAL PHOSPHORIC ACID.

Determine total phosphoric acid in the three samples by the following methods:

(1) Official gravimetric method (Bulletin 46, revised edition, Division of Chemistry, U. S. Department of Agriculture, p. 12).

(2) Volumetric method: Make solution of the phosphate according to one of the official methods. Take an aliquot portion containing not more than 30 mg P_2O_5 , add ammonium hydrate in excess, then nitric acid in slight excess, then an excess of the official molybdate solution. Place in a water bath at 45° to 50° C. for one-half hour with occasional stirring, filter, and wash with water until 25 cc of the filtrate fails to neutralize one drop of the standard potassium hydrate solution. Transfer filter and contents to the beaker. Add about 30 cc of water and sufficient standard alkali to dissolve the yellow precipitate. Titrate the excess of alkali with standard acid, using phenolphthalein as indicator. (See Bulletin 46, Division of Chemistry, U. S. Department of Agriculture, p. 13.)

CITRATE SOLUBLE PHOSPHORIC ACID.

Determine available phosphoric acid in the sample of basic slag (No. 1) as follows:

Place 3 grams of the slag in a 300 cc flask, add 200 cc of 1 per cent solution of citric acid, place in a water bath, and maintain at 50° C. for one hour. Shake vigorously five times during the digestion at intervals of about ten minutes, cool under faucet, make up to mark with water, filter out 50 cc; to this add ammonium hydrate until alkaline, then nitric acid until slightly acid, and about 10 grams of ammonium nitrate. Place in a water bath at 75° C. Add 75 cc molybdate solution, let stand thirty minutes with frequent stirring, cool, filter, wash with cold water, and proceed as under total phosphoric acid. (Bulletin 46, Division of Chemistry, U. S. Department of Agriculture, p. 12.)

IRON AND ALUMINA.

Determine iron and alumina in the three samples by the following methods:

(1) *Acetate method.*

Take 50 cc of the solution corresponding to 0.5 gram of substance; add ammonium hydrate until slightly alkaline. Add hydrochloric acid drop by drop until the precipitate just dissolves, then 2 or 3 drops more, heat to boiling, and add an excess of ammonium oxalate. Let stand until cold, filter, and wash with hot water. Treat the filtrate with ammonium hydrate drop by drop until a precipitate begins to form and then add 25 cc of a 25 per cent ammonium acetate solution. Heat to boiling, filter, and wash with a 5 per cent ammonium nitrate solution. Dissolve the residue in nitric acid, add a few drops of sodium phosphate and then ammonium hydrate until a precipitate begins to form. Treat with an excess of ammonium acetate, heat to boiling, filter, wash with ammonium nitrate, dry, ignite, and weigh as iron and aluminum phosphates.

(2) *Molybdate method.*

(a) To 50 cc of the solution, corresponding to one-half gram of substance, add ammonia until alkaline, make slightly acid with nitric acid, heat to 50° C, and add slowly an excess of molybdate solution, stirring constantly. Let stand until cold, filter, and wash with cold water.

(b) Treat the filtrate obtained under (a) with ammonium hydrate until nearly alkaline, add an excess of ammonium sulphid, heat to 80° C, let stand three hours, filter rapidly under pressure, and wash five or six times with water containing ammonium sulphid. Dissolve the residue in dilute nitric acid, boil a few minutes, add ammonia until slightly alkaline, and determine the iron and alumina as usual.

(c) Proceed as under (a) and to the filtrate add ammonium hydrate in slight excess. Heat to 50° C. and let stand for thirty minutes, filter, and wash with hot water. Dissolve the residue in hydrochloric acid, add ammonium hydrate until alkaline, charge the solution with excess of hydrogen sulphid, and add dilute hydrochloric acid until decidedly acid. Let stand a few minutes, filter, and wash with 1 per cent hydrochloric acid, then add a few drops of nitric acid to filtrate (which should be clear), boil to render iron in ferric condition, and determine iron and alumina in the usual manner.

In case it is found impracticable to do all the work indicated the referee would be glad to have at least the determination of iron and alumina made in Nos. 2 and 3 by the methods given. Please report results thirty days previous to the next meeting of the association.

H. K. MILLER, *Referee.*

C. H. JONES, *Associate Referee.*

Results were obtained from five laboratories, including the work of eight analysts. These results appear below in Tables I and II. Before examining the results it may be well to explain briefly the points which the referee had in mind when preparing the instructions. At the last meeting of this association a resolution was passed requesting the referee for 1901 to make a study of basic slag with a view to finding a suitable method for estimating its commercial value. Accordingly, a sample of basic slag was sent out with the instructions for a trial method for approximating its available phosphoric acid. Aside from this, and a further test of the volumetric method as proposed by Runyan, it was thought advisable to make an effort to find a satisfactory method for estimating iron and alumina in the presence of phosphoric acid. Samples Nos. 2 and 3 were prepared with special view to this line of work. While many believe a satisfactory solution of this problem is to be found in some modification of the acetate method, others have been unable to find any encouragement through its use. The methods sent out by the referee are simply modifications of methods formerly proposed. Results obtained by two other modifications of the acetate method were reported and are included in the results. Table I contains the results obtained on total phosphoric acid and the results on the phosphoric acid in the Thomas slag soluble in a 1 per cent citric acid solution.

TABLE I.—*Results on phosphoric acid.*

Analyst.	Volumetric method.			Gravimetric method.			Citric acid.
	No. 1.	No. 2.	No. 3.	No. 1.	No. 2.	No. 3.	
F. B. Carpenter, Richmond, Va				18.81	35.98	20.33	
G. A. Hanvey, Richmond, Va	18.55	35.95	20.33	18.66	35.90	20.37	
E. G. Runyan, Washington, D. C	18.03	35.07	19.90	17.84	35.10	19.94	6.08
J. G. Heavener, Richmond, Va					^b 35.60		
		35.73		^b 18.39	^b 35.60		
	^a 18.46	35.61		^b 18.43	^b 35.60		
	^a 18.39		19.91	^b 18.39	35.60		
		35.76	19.91		35.55		
	^a 18.47	35.77		^c 18.33			
	^a 18.46			^c 18.33	^a 35.60		
					^a 35.58		
B. F. Robertson, Clemson College, S. C.	18.47	35.40	20.01	18.65	35.67	20.13	
						20.32	6.75
C. C. McDonnell, Clemson College, S. C.	18.32	35.58	20.50	18.62	35.64	20.10	
						20.41	6.82
H. K. Miller, Lake City, Fla		35.80					
	18.20	35.70		18.26	35.63		
	18.40	35.60		18.34	35.65		
A. W. Blair, Lake City, Fla	18.85	36.05					
	19.05	35.90					
	19.02	36.07	20.57				
	19.07	36.07	20.55	18.85	35.69	20.30	6.79
	18.68	35.70	20.62	18.75	35.74	20.29	6.73
	18.80	36.00	20.50			20.44	6.78
	18.82	35.77	20.55			20.58	6.79
	18.85	35.72	20.55				

^aPrecipitated at room temperature.^bDissolved in sulphuric acid.^cSilica removed.

Table II contains the results obtained on the iron and alumina in samples Nos. 2 and 3.

TABLE II.—*Results on iron and alumina.*

Analyst.	Carpenter's acetate method.		Referee's acetate method.		Molybdate method B.		Molybdate method C.	
	No. 2.	No. 3.	No. 2.	No. 3.	No. 2.	No. 3.	No. 2.	No. 3.
E. G. Runyan, Washington, D. C.	*3.70	*4.38	2.80	2.58	7.20	7.85	2.50	4.33
F. B. Carpenter, Richmond, Va.	3.02	4.63						
G. A. Hanvey, Richmond, Va.	2.95	4.50						
S. H. Sheib, Richmond, Va.	2.83	4.53						
C. C. McDonnell, Clemson College, S. C.			2.30	4.20			2.50	4.11
B. F. Robertson, Clemson College, S. C.			2.98	4.06	2.77	3.51		
A. W. Blair, Lake City, Fla.			2.64	2.22				
.....		4.04	2.38	3.69				
.....		3.95	2.44	2.84				
.....				1.48				
H. K. Miller, Lake City, Fla.					6.68	6.74		
.....					6.28	6.82		
Actual		4.51		4.51		4.51		4.51

* Acetate method (Bull. 62, Div. Chem., p. 35).

NOTES BY THE ANALYSTS.

F. B. Carpenter.—In the volumetric method we used a temperature of from 60° to 65° C., allowing the solution with the precipitate to remain in the bath about twelve minutes. All the methods for iron and alumina were tried, but none proved satisfactory. In the case of the basic slag the results do not agree as well as with the other samples, and we had the same experience last year with material of this kind. I think that the trouble probably lies in the fact that there is a large amount of soluble silica present, which, in case of the gravimetric method, may be precipitated to some extent along with the magnesia mixture.

E. G. Runyan.—In some cases the determinations were made in sextuplicate and in no case less than duplicate. Judging from the agreement of the results the figures on phosphoric acid are fairly satisfactory. The results on iron and alumina are rather wild, but I am not willing to admit that this is entirely the fault of the analyst. I believe the methods proposed by the referee are faulty, as they embrace steps which McElroy, Krug, and others in this laboratory have shown to yield unreliable results. With each method the determinations were made in duplicate and the averages given in the table represent a fair trial of the proposed method.

J. G. Heavener.—The white precipitate of sample No. 1 still had traces of iron after two precipitations.

A. W. Blair.—It was found impossible to obtain a perfect solution of No. 1 by the usual methods employed. It was necessary to dissolve on the water bath in order to prevent the formation of a gelatinous precipitate, which always interfered with a perfect solution, inasmuch as it adhered to the bottom of the flask when heated on a hot plate.

B. F. Robertson.—The ferric oxid from the ammonium sulphid precipitate contained some manganese, which was separated by dissolving in hydrochloric acid and precipitating the ferric oxid with ammonium acetate.

C. C. McDonnell.—The iron and alumina precipitate from No. 1 (molybdate method) contains some manganese, which accounts for the alumina being so high. In

this determination also there was a loss of iron due to the fact that on acidifying the sulphids with hydrochloric acid some sulphid of iron was left undissolved. The results on phosphoric acid are averages of several determinations. The results with the volumetric method show wide variations in some cases.

DISCUSSION OF RESULTS ON PHOSPHORIC ACID.

On examining the results by the volumetric method on sample No. 1 it will be seen that rather wide variations occur in one or two cases. The results are too low in a few cases, and I think it can be safely assumed that this is due to imperfect solution and not to the volumetric method proper. The results reported by one analyst seem rather high. Altogether, with these exceptions, the results given are quite satisfactory. I believe the same trouble has been experienced with sample No. 2. A few of the results are somewhat low; only one, however, is too low to be included in the averages. I am inclined to believe the high results represent more nearly the actual phosphoric acid present in samples Nos. 1 and 2 than the average. It has been my experience that it frequently occurs in dissolving certain phosphates and Thomas slag that solution is not complete even after prolonged digestion with fresh additions of acid. It has been shown, too, that during the digestion it sometimes occurs that the dissolved phosphate becomes caked on the side of the flask above the surface of the acid and is thus rendered insoluble. In the case of Thomas slag I have been unable to get a perfect solution when strong acids were poured on the material and the flasks placed on the hot plate. The variations obtained on No. 3 by the volumetric method are only slight, except in one or two cases. Passing to the gravimetric method we find in the case of No. 1 that the variations are greater than in the case of the volumetric method. With No. 2 by this method it will be seen that the greatest variations occur. The result on No. 3 by the gravimetric method are in one or two cases a little wild, but on the whole are fairly satisfactory. From the results obtained last year and those of this year, and from my experience with the modification of the volumetric method as proposed by Runyan, I think it is capable of giving highly satisfactory results and of being expeditiously handled. The phosphoric acid in sample No. 1, soluble in a 1 per cent citric acid solution, is reported by four analysts with fairly concordant results.

DISCUSSION OF RESULTS ON IRON AND ALUMINA.

As usual few results were reported on iron and alumina, and, as is generally the case, quite varying results have been obtained. The results on No. 1 have not been included in the table, inasmuch as the manganese present interfered with the determination of iron and alumina and those reported were exceedingly wild, varying from 3 to 20 per cent. The results obtained on Nos. 2 and 3 by the acetate method as modified by Carpenter are quite satisfactory compared with previous reports. It will be seen from the few results reported that the referee's proposed acetate modification does not yield good results. This is true also of the molybdate method B, but the molybdate method C yielded fairly concordant results. It is not possible to draw any conclusion from the results obtained on the iron and alumina, inasmuch as the data is too meager.

RECOMMENDATIONS.

It would evidently be unwise to make any recommendations based on the results which have been reported on these samples. It is quite evident, however, that one question should receive some action by this association, and that is a method applicable to Thomas slag. While I am not prepared to make a direct recommendation in regard to this, I think it would be well for the committee to consider the advisability of adopting some form of the citric acid method. The Wagner method, I think, comes in for the most favorable consideration. It must be borne in mind that "available phosphoric acid" is an unwise term, and I think it would be well to

have in view the idea of valuing fertilizers according to the constituents found therein. This applies especially to phosphoric acid, and inasmuch as we have several materials containing phosphorus, but in different combinations, which are useful as fertilizers, it is not probable that a single method will be found applicable to all these. The only solution apparent at this time is to recognize this difference and apply special methods.

The PRESIDENT. Are there any other papers to be presented in relation to phosphoric acid—any remarks or discussion?

Mr. HUSTON. Are there any specific recommendations in this report?

The PRESIDENT (to Mr. Wiley). What is the particular point with reference to any recommendations of a general kind in the report?

Mr. WILEY (reading from the recommendations of the referee for the information of Mr. Huston). "While I am not prepared to make any direct recommendations," etc.

Mr. HUSTON. Although there are no direct recommendations, the report is to go to the committee, as there may be some points in it that we might take up. We tried this course at least ten years ago and I do not see the use of trying the same thing over and over again. If we take it up at all we must proceed on an entirely different basis. I do not think it difficult to determine the amount of iron in these samples. It can be done without trouble. I have had very excellent results on iron, and there was no difficulty in handling the alumina, but I think we ought to proceed on an entirely different basis. I appreciate the fact that a majority of the former members of this association were anxious to have iron and alumina included, but even if they were trying to follow that method I think we ought to take a new start and see if we can not get hold of some method, even if it is a long one, which will finally determine these points; but the method will have to be wholly different from any we have been using so far. I think it can be done, but I do not think we are on the right track.

The PRESIDENT. Is the committee which was appointed to consider and report upon changing the number of committees on recommendations of referees ready to report? If so, the report will be received at this time.

REPORT OF COMMITTEE ON CHANGING NUMBER OF COMMITTEES ON RECOMMENDATIONS OF REFEREES.

The committee to whom was referred for consideration and report the question of the number of committees on recommendations desirable for our present work would recommend that, instead of one committee, as heretofore, three committees of five members each be appointed, with the following assignment of subjects to each:

- A. Phosphoric acid, potash, soils, ash, and insecticides.
- B. Dairy products, foods and feeding stuffs, sugar, tannin.
- C. Liquors and detection of food adulteration.

WILLIAM FREAR,
H. J. WHEELER,
B. W. KILGORE,

Committee.

Mr. MAY. I move the adoption of the report.

The PRESIDENT. A motion has been made and seconded that the report of the committee as read be adopted. Discussion is in order. The report is adopted.

The report of the referee on soils is next in order.

Mr. WILEY. Mr. Jaffa unfortunately can not be here, but he has sent his report, which is ready for presentation. This report begins with a statement of the work to be done.

REPORT ON SOILS.

By M. E. JAFFA, *Referee*.

The work of the referee on soils was confined this year to the further investigation of methods for obtaining the available or assimilable plant food. The analytical data here reported are gained from five soils—two from California and three from different sections of the Eastern States—the object being to gather data from as many different kinds of soils as possible. Credit is due Mr. C. A. Triebel for material assistance in the chemical work here reported.

The circular letter given below was sent to the different experiment station chemists asking for cooperation in the work for the year. Thirteen favorable replies were received, but analytical data have been received from only three stations, ^a viz, Oregon, Ohio, and Virginia. It is to be exceedingly regretted that more results are not at hand for comparison.

MARCH 23, 1901.

DEAR SIR: The referee on soils of the Association of Official Agricultural Chemists this year will continue the study of methods for the determination of available mineral plant food; also the question of humus and humus nitrogen. We would like to have your cooperation in the work. Please notify the referee if you will undertake it.

M. E. JAFFA, *Referee*.

F. P. VEITCH, *Associate Referee*.

The working directions which were sent to the respondents to the circular letter are as follows:

DIRECTIONS FOR WORK ON A. O. A. C. SOIL SAMPLES FOR 1901.

I.—THE DETERMINATION OF MOISTURE.

Use the official method described in Bulletin 46, page 71, Division of Chemistry, U. S. Department of Agriculture. Calculate all results to the water-free basis.

II.—THE DETERMINATION OF AVAILABLE POTASH SOLUBLE IN N/5 HCl.

(a) *Preliminary treatment*.—Digest 20 grams of soil with 200 cc of N/5 HCl, in a water bath, at 40° C., for five hours, shaking every half hour. Titrate 20 cc of the clear filtrate against a standard KOH solution, using phenolphthalein for the indicator. From the data thus secured calculate the amount of HCl necessary to be added so that the solution will be N/5 after allowing for the acid neutralized.

(b) *The determination*.—Two thousand to 3,000 cc of acid, corrected for neutralization, as directed under (a), are heated in a stopped receptacle to 40° C. Ten grams of soil for every 100 cc of acid are now added and the digestion continued at this temperature for five hours, shaking thoroughly every half hour. The contents are then shaken and filtered through a dry, ribbed filter of two thicknesses of paper, refiltering the first portion of the filtrate if cloudy. Take an aliquot portion of the filtrate, evaporate to a small volume in a porcelain dish, transfer to a platinum dish, add a little sulphuric acid, and determine the potash by the Lindo-Gladding method.

^a Subsequent to the reading of the report, data were received from two additional sources, i. e., Kentucky Agricultural Experiment Station and Division of Soils, U. S. Department of Agriculture, Washington, D. C.

III.—THE DETERMINATION OF AVAILABLE POTASH AND PHOSPHORIC ACID SOLUBLE IN 2 PER CENT CITRIC ACID SOLUTION.

(a) *Preliminary treatment.*—Determine, in separate portions of the soils, the amount of carbonic acid. From the data thus obtained calculate the amount of citric acid necessary to be added so that the solution will be 2 per cent after allowing for the carbonic acid expelled.

(b) *The determination.*—Digest 50 grams of soil with 300 cc of 2 per cent citric acid solution, with frequent shakings, for twenty-four hours. Filter through two thicknesses of paper. Evaporate filtrate to a small volume in a porcelain dish, transfer to a platinum dish, evaporate to dryness, carefully ignite the residue, digest with hydrochloric acid and water (sol. A).

Take two aliquot portions of the sol. A. Use one portion for determining phosphoric acid according to one of the official methods. Evaporate the other portion to a small volume, if necessary, in a porcelain dish, transfer to a platinum dish, add a little sulphuric acid, and determine the potash by the Lindo-Gladding method.

IV.—HUMUS AND HUMUS NITROGEN.

Determine humus and humus nitrogen in accordance with directions given on page 76, Bulletin 46, revised edition, Division of Chemistry. In addition thereto transfer an aliquot of the ammoniacal humus solution, prepared as directed, to a flask suitable for Kjeldahl digestion, evaporate to dryness on water bath and then expose to temperature of 100° C. in order to drive off the *free* ammonia. Treat the residue thus obtained with MgO, and thus determine the amount of *combined* ammonia.

Transfer another aliquot of the ammoniacal humus solution to a Kjeldahl flask, evaporate as above, and determine the total nitrogen in the residue by the Kjeldahl method. The question is, does the nitrogen obtained by treatment with NaHO solution bear any relation to the amount of nitrogen contained in the residue from the ammonia treatment, after the combined ammonia (expelled by MgO) has been deducted therefrom?

DESCRIPTION OF THE SOILS.

No. 1.—“Adobe soil” from the moist lands bordering on Chino Creek, Los Angeles County, Cal., taken to depth of 12 inches. A mouse-colored, moderately clayey loam, somewhat silty; the dry lumps crush quite readily between the fingers and show little or no grit. With acid there is effervescence on wetting, the color deepens considerably, and when kneaded the soil becomes quite adhesive, showing that it must not be tilled while wet. Its natural vegetation is a dense, tall growth of grass, with some spots bearing the yerba mansa (*Anemopsis californica*) and sunflower. The complete analyses are as follows:

	Per cent.
Coarse material above 0.5 mm in diameter.....	10.00
Fine earth	90.00
Total	100.00

Chemical analysis of fine earth.

	Per cent.
Insoluble matter	62.62
Soluble silica.....	8.30
	70.92
Potash (K ₂ O)	.95
Soda (Na ₂ O)	.50
Lime (CaO)	5.07
Magnesia (MgO)	.84
Trimanganese tetroxid (Mn ₃ O ₄)	.06
Ferric oxid (Fe ₂ O ₃)	6.43
Alumina (Al ₂ O ₃)	4.88
Phosphoric acid (P ₂ O ₅)	.21
Sulphuric acid (SO ₃)	.06
Carbonic acid (CO ₂)	3.76
Water and organic matter.....	6.02
Total	99.70

Mechanical analysis.^a

	Per cen
Colloid clay, per cent.....	13.44
Silt:	
0.25 mm, hydraulic value	20.65
0.25 mm, hydraulic value.....	1.37
0.50 mm, hydraulic value.....	4.91
1 mm, hydraulic value	5.95
2 mm, hydraulic value.....	13.46
4 mm, hydraulic value.....	10.36
8 mm, hydraulic value.....	9.25
16 mm, hydraulic value.....	12.01
32 mm, hydraulic value.....	2.75
64 mm, hydraulic value.....	1.66
	95.81
Water capacity.....	60.20

No. 2.—Orange-tinted loam from Arlington Heights, San Bernardino County, Cal. The soils of this tract are of great depth; in the breaks profiles of as much as 30 feet of sensibly uniform red loam may be seen, and it will be noted by reference to the following analyses that it is of almost uniform composition, chemically, to the depth of 10 feet.

The roots of wild shrubs are found within it to depths of from 6 to 8 feet, showing it to be as easily penetrable by roots as it is by water, its red tint alone being proof of its perfect drainage.^b Vegetation: Cactus, baeria, croton, and greasewood.

Chemical analyses of soil and subsoil of Arlington Heights.^a

	Soil No. 2.	Subsoil No. 2.
Depth	1	10
	<i>Per cent.</i>	<i>Per cent.</i>
Coarse material, > 0.5 mm.....	20.00	23.00
Fine earth	80.00	77.00
	100.00	100.00
Insoluble matter and soluble silica	^b 84.61	^c 83.51
Potash (K ₂ O).....	.87	.93
Soda (Na ₂ O)	.29	.36
Lime (CaO)	1.57	1.73
Magnesia (MgO)	1.33	1.58
Trimanganese tetroxid (Mn ₃ O ₄)	.04	.04
Ferric oxid (Fe ₂ O ₃)	4.20	5.34
Alumina (Al ₂ O ₃)	5.30	5.08
Phosphoric acid (P ₂ O ₅).....	.14	.16
Sulphuric acid (SO ₃)		
Water and organic matter	1.60	1.44
	99.85	100.17
Hygroscopic moisture absorbed at 15° C	1.77	2.16

^a Cal. Sta. Report, 1890, p. 41.^b Insoluble matter, 76.41; soluble silica, 8.20.^c Insoluble matter, 75.44; soluble silica, 8.07.

Soils Nos. 1 and 2 were selected for distribution because they are both virgin, productive soils, showing high percentages of potash and phosphoric acid, but decidedly

^a Dr. R. H. Loughridge, analyst.^b Cal. Sta. Report, 1890, p. 39.

different in character and humus content, No. 1 being rich and No. 2 poor in this respect.

Soils Nos. 3, 4, and 5 were kindly furnished by Mr. B. L. Hartwell, of the Rhode Island station. The field records of these soils will be found in the Proceedings of the Seventeenth Annual Convention of the Association of Official Agricultural Chemists, Bulletin 62, Division of Chemistry, United States Department of Agriculture, pages 75-77.

The percentages of fine and coarse earth, as obtained by Mr. Hartwell, in soils Nos. 3, 4, and 5 are given in the subjoined table:

	No. 3. Wapping soil.	No. 4. Lime rock soil.	No. 5. Amherst soil.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Coarse earth, >0.5 mm	16.50	13.90	2.40
Fine earth.....	83.50	86.10	97.60
Total	100.00	100.00	100.00

The fine earth in each case was distributed, as it is this part of the soil that forms the storehouse of available plant food.

POTASH.

It was desired this year to make further studies of the citric acid and N/5 hydrochloric acid methods for determining the amount of available potash and phosphoric acid contained in soils, more particularly as in the case of Nos. 3, 4, and 5 we had the data^a obtained by Mr. Hartwell in his past experiments in those soils for reference and comparison.

Unfortunately we have far too few results to enable us to draw any conclusions whatever.

It appeared to the referee that more extended work on the citric acid method was called for. Citric acid certainly approaches more nearly to the root acid than any of the mineral acids, and also cold digestion is closer in touch with the processes of nature than is a temperature of 40° C.

The citric acid method is the one in use at the California station, and the results agree satisfactorily with actual practice. Dr. Dyer recommends a 1 per cent solution of citric acid; but, owing to the generally high content of lime in California soils and to the fact that the acidity of citrus roots is quite often over 1 per cent, it was deemed advisable to use a 2 per cent solution, after allowing for the carbonate of lime which the soil might contain.

TABLE I.—*Potash (K₂O) soluble in N/5 HCl.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. W. Ames, Ohio.....		0.0299	0.0110	0.0155	0.0175
Ellett and Gibboney, Virginia.....	0.1231	.0284	.0150	.0119	.0124
Major Edwards, Oregon	.0830	.0610	.0280	.0310	.0360
M. E. Jaffa, California.....	.1149	.0298	.0205	.0174	.0201
F. P. Veitch, Washington, D. C. ^b	.0938	.0139	.0242	.0311	.0155

^a U. S. Dept. of Agr., Bureau of Chem. Bul. 62, p. 73.

^b These data were received too late to be included in the discussion of results and general averages, but are added to the table.

DISCUSSION OF RESULTS.

An examination of Table I shows soil No. 1 to be very rich in available potash, and the results obtained by the Virginia and California stations agree quite closely; similar conditions obtain for soil No. 2, if we leave out of consideration the data obtained by the Oregon station. The figures given for Nos. 3, 4, and 5 are not very satisfactory.

The maxima per cents are quoted by the Oregon station in each case, being, respectively, 0.028, 0.031, and 0.036. The minimum for the soil No. 3 is 0.011 per cent, to the credit of the Ohio station, and the minima per cents for Nos. 4 and 5, 0.0119 and 0.0124, respectively, are given by the Virginia station. The average per cents of all the determinations are as follows:

No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
0.107	0.0373	0.0186	0.0189	0.0215

If we omit the figure for No. 2, obtained by the Oregon station, the average will then be 0.0294, which would seem to be more correct for the average than the previous one.

It is a matter of regret to the referee that he can not present more data on the citric acid method than those which are given in the following table:

TABLE II.—*Potash (K_2O) soluble in 2 per cent citric acid.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Major Edwards, Oregon.....	0.083		0.014	0.018	0.023
M. E. Jaffa, California.....	.056	0.017	.016	.014	.017

The experience of the California station has shown that the results yielded by this method are always less than the corresponding ones reported for the hydrochloric acid treatment. The figures given in Table II are in accordance with our other experiments in this direction.

The above data are far too meager to allow of summarizing in any definite way. But if a tentative conclusion could be ventured we might say, in accordance with our experience in California and the results as shown by the careful and able experiment^a of Mr. Hartwell, that any soil showing 0.02 per cent of K_2O by either of the above methods is not in pressing need of potash fertilization. More work along this line is urgently called for. The citric acid method certainly should not be discarded without proper and careful investigation.

PHOSPHORIC ACID.

In Table III are given the results obtained for phosphoric acid by the citric acid digestion.

TABLE III.—*Phosphoric acid soluble in 2 per cent citric acid.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Major Edwards, Oregon.....	0.012	0.019	0.025	0.018	0.023
M. E. Jaffa, California.....	.031	.027	.033	.016	.024
F. P. Veitch, Washington, D. C. ^b	.181	.055	.133	.044	.021

^aU. S. Dept. Agr., Bureau of Chem. Bul. 62, p. 73.

^bData received too late to be included in discussion of results or in the general averages, though included in the table.

Again, we find that there has not been sufficient cooperation to warrant our forming any conclusions; and in view of the importance of adopting some method for determining the available plant food in soils, one which will yield concordant results in the hands of expert chemists, and also because so little cooperation is afforded the referee on soils each year, I would offer the following suggestion: That a large committee of chemists be appointed, with the referee on soils as chairman, to investigate and report upon some method for determining the available plant food in soils in place of continuing the practice now in vogue.

HUMUS AND HUMUS NITROGEN.

The object of the work along this line was to ascertain, if possible, the relation, if any, which exists between the nitrogen extracted by ammonia and that contained in the sodium hydrate or potassium hydrate leachings; but, as evidenced by the tables below, far too few data have been gathered to enable us to arrive at any conclusions.

In Table IV are given the results obtained for humus:

TABLE IV.—*Humus in soils.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Ellett and Gibboney, Blacksburg, Va.....	2.52	0.81	4.24	2.12	3.80
Major Edwards, Corvallis, Oreg.....	2.60	.89	2.45	2.02	2.04
M. E. Jaffa, Berkeley, Cal.....	2.14	.42	2.44	2.03	2.04

The discrepancies here shown are far from satisfactory, and the same may be said with respect to Table V, showing percentages of nitrogen in residue from ammonia leachings:

TABLE V.—*Total nitrogen in residue.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Ellett and Gibboney.....	0.437	0.203	0.448	0.455	0.466
M. E. Jaffa.....	.228	.084	.232	.171	.202

The above differences are somewhat difficult to account for in view of the fact that the figures for the nitrogen as combined ammonia in the same residue do not show similar discrepancies. This is seen by a glance at the following statement:

TABLE VI.—*Nitrogen, ^a as combined ammonia in residue from ammonia leachings.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Ellett and Gibboney.....	0.053	0.031	0.089	0.052	0.159
M. E. Jaffa.....	.054	.026	.073	.067	.073

^a Determined by treatment with magnesium oxid.

Subtracting the respective results contained in Tables V and VI, and arranging, we have the following:

TABLE VII.—The “organic” nitrogen in the residue from ammonia leachings.

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Ellett and Gibboney	0.384	0.172	0.359	0.403	0.307
M. E. Jaffa	.174	.058	.159	.104	.129

Comparing the figures in Table VII with the data reported for the humus nitrogen dissolved by the sodium hydrate solution, we have:

TABLE VIII.—Comparison of humus nitrogen extracted by ammonia and sodium hydrate solution.

Analyst.	No. 1.		No. 2.		No. 3.		No. 4.		No. 5.	
	Ammo- nia.	Sodium hy- drate.	Ammo- nia.	Sodium hy- drate.	Ammo- nia.	Sodium hy- drate.	Ammo- nia.	Sodium hy- drate.	Ammo- nia.	Sodium hy- drate.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
Ellett and Gibboney	0.384	0.173	0.172	0.043	0.359	0.065	0.403	0.096	0.307	0.106
Major Edwards		.141		.061		.147		.145		.078
M. E. Jaffa	.174	.171	.058	.059	.159	.160	.104	.117	.129	.132
Average		.162		.054		.124		.119		.105

The above is not a very satisfactory showing. While there is agreement between some of the results reported for the nitrogen by sodium hydrate, the percentages of this element extracted by the ammonia solution are far from being concordant.

Again, the data presented by the California station would indicate that there is a relation between the nitrogen dissolved by sodium hydrate and that by ammonia, and, further, that after deducting the amount of nitrogen in the form of combined ammonia, the percentages of organic nitrogen by treatment with sodium hydrate and ammonia, respectively, are practically identical in Nos. 1, 2, 3, and 5. The presentation by the Virginia station, however, shows no such relation, the amount of organic nitrogen by the ammonia treatment far exceeding that by sodium hydrate.

The determinations of humus nitrogen by sodium-hydrate digestion were checked by similar treatment with 5 per cent potassium hydrate solution, with the result as shown in Table IX, which also contains the complete data obtained by the California station for humus and humus nitrogen.

TABLE IX.—Humus and humus nitrogen.

No.	Humus.	Humus nitrogen in soil.					Humus nitrogen in humus.			
		Nitrogen as com- bined am- monia.	Total nitrogen in hu- mus.	“Organ- ic” ni- trogen.	Nitrogen in KOH leach- ings.	Nitrogen in NaOH leach- ings.	Nitro- gen as com- bined NH ₃ in humus.	Organic nitro- gen in humus.	Nitro- gen in KOH leach- ings.	Nitro- gen in NaOH leach- ings.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1	2.14	0.054	0.228	0.174	0.177	0.171	2.52	8.13	8.25	8.00
2	.42	.026	.084	.058	.062	.059	6.19	13.83	14.76	14.05
3	2.44	.073	.232	.159	.162	.160	3.00	6.51	6.64	6.56
4	2.03	.067	.171	.104	.114	.117	3.30	5.12	5.62	5.76
5	2.04	.073	.202	.129	.132	.132	3.58	6.32	6.47	6.47

An examination of this table proves that the amounts of nitrogen dissolved by potassium hydrate and sodium hydrate, respectively, are identical, and also that while the percentage of humus in Nos. 3, 4, and 5 are high, that of the humus nitrogen is correspondingly low.

In conclusion, the referee would recommend that extended investigation be carried on along this line of work, but, as previously suggested, owing to the lack of sufficient cooperation, more profitable results would in all probability be obtained if the subject were left for consideration to a committee with the referee on soils as chairman.

ROTHAMSTED METHOD OF SAMPLING SOILS.

In accordance with the recommendations made at the last meeting of the association, the referee submits the following on this subject. The writer is in heartiest sympathy with Dr. Dyer in his desire to obtain a uniformity of method for the sampling of soils, and feels with him that the importance of the subject warrants continued and determined effort to accomplish that end.

The taking of the sample is one of the most vital parts of soil investigation, and we can never arrive at accurate and uniform results until the first and crucial step in the process has been thoroughly discussed and the best method suitable for all places and conditions decided upon.

The method^a as practiced at Rothamsted is as follows:

The following plan has been used in Rothamsted since 1856. A frame made of stout sheet iron, in shape a rectangular prism, open at top and bottom, is driven into the soil by repeated blows of a wooden rammer, till the soil has the same level inside and outside the frame. The soil inside the frame is then cut out and constitutes the sample of the first depth or surface soil. That the frame is accurately emptied is ascertained by trials with a wooden gauge of the same depth as the iron frame. If a sample of the next depth is to be taken, the soil is cleared away around the outside of the frame till the level is reduced to that of the bottom of the frame; the frame is then driven down again and the former operations are repeated.

Soil sampling at Rothamsted is usually carried down to three depths, but in a good many cases it has been carried down to twelve depths. The area of the sampling frame used for the first depth is usually 144 square inches (12 by 12 inches), a smaller frame, 6 by 6 inches, being used for the succeeding depths. The depth of each frame is 9 inches. Messrs. Lawes and Gilbert are of the opinion that 6 inches would have been a better depth to adopt for the samples of surface soil, but so much work has been done with 9 inches that it would now be very inconvenient to make any alteration.

The iron frame has a stout rim along its upper edge to increase its strength. The best sampling frame is made of cast steel; this form of frame needs no rim. Models both of the larger and smaller steel frames used at Rothamsted are before you, and I am instructed by Sir John Lawes to leave these models with your Department of Agriculture.

When the soil sampling is carried below the first depth care must be taken when digging around the frame that each depth of soil removed is placed by itself, so that when the pit is filled in the soil may be returned to its proper position. A record must be kept of the place where the sampling was conducted, as a soil can not be accurately sampled twice in the same place.

Each sample of soil is weighed as soon as it is removed from the frame, and is put into a bag by itself. When the soil reaches the laboratory it is at once broken up by hand into small pieces and laid on paper trays, which are placed on the shelves of a storeroom kept at a temperature of about 55° C. till thoroughly dry; each sample is then returned to its bag. This immediate drying of the soil at a low temperature is essential, if changes in the organic matter, and especially nitrification, are to be stopped. This practice dates at Rothamsted from 1877. After drying, the soil may be stored till leisure is found for further work. Each bag is then weighed. The soil is crushed and passed through a one-fourth inch sieve; the stones that do not pass through this sieve are weighed as stones. All that passes through the sieve is thoroughly mixed and a sufficient quantity is finely powdered for analysis. Mixed samples are prepared after the soil has passed through the one-fourth inch sieve, or after it has reached the stage of fine powder.

^a U. S. Dept. Agr., Office of Expt. Sta. Bul. No. 8, p. 39.

Dr. Dyer states:

This method of taking soil samples, with the exception of the adoption of the definite temperature above mentioned for drying, has been, as has already been said, in use in Rothamsted since 1856, and it is interesting here to record that between 4,000 and 5,000 individual samples of the Rothamsted soils have been collected in the fashion above described.

In view of these facts it can be easily understood why this plan of sampling should not be changed for Rothamsted. But in a new country it is an entirely different question, and we ought to adopt some uniform method of sampling soils. It seems to the writer, however, that the Rothamsted method has several objections outlined below:

- (1) The sampling tool, as described, is much too heavy for rapid work.
- (2) When a virgin soil is sampled more than the requisite quantity of soil is collected.
- (3) If a fertilized field is in question, then more samples should be gathered and the average taken. This with the Dyer method would entail a large amount of work.

(4) It is almost impossible by this method to obtain an accurate sample of the surface soil unless it should be just 8 or 9 inches in depth.

Some surface soils are 6 inches, others 12 inches in depth, and then again, as in some localities of California, the surface soil is much deeper.

Such a practice as described by Dr. Dyer would utterly fail of its purpose if applied to the larger part of the arable lands of California.

It would seem that the post-hole auger, which has been used with success in this State since its adoption by Dr. Hilgard, is much better adapted to the sampling of soils than is the implement suggested by Dr. Dyer, because—

- (1) Less work is required in taking the sample.
- (2) More samples can be collected in a given time.
- (3) The exact point of change from surface to subsoil can be ascertained. No arbitrary depth can be, or should be, assigned for surface soil. It depends upon the depth of the humus layer; that and that only should, as a rule, constitute the surface soil. When, however, this layer is only about 3 inches deep, then the case is somewhat different and at least 6 inches should be taken.
- (4) Much less bulk has to be handled.

The foregoing are suggestions offered for the consideration of the association.

It must be remembered that at the meeting three years ago a conclusive report was rendered on the question of a uniform fertilizer-control law by a committee composed of some of the most prominent members of the association. In view of the manifest necessity of a rational system of sampling of soils and of the adoption of a method for determining their available plant food, the referee suggests that a similar committee be appointed for the consideration of these subjects.

MR. WITHERS. I have a report on soils which I would like to present to this committee.

THE PRESIDENT. If the report is on soils it may be read at this time.

THE NITRIFICATION OF AMMONIUM SULPHATE AND COTTON-SEED MEAL IN DIFFERENT SOILS.

By W. A. WITHERS and G. S. FRAPS.

In Bulletin No. 176 of the North Carolina Agricultural Experiment Station, some account of which was read before this association at its meeting in 1900, the authors reported the results obtained by making determinations of the amounts of nitrates

present in a soil with which different samples of various nitrogenous materials had been in contact for about three weeks, in some cases with and in others without the addition of calcium carbonate. It was found, as a result of that series of experiments, that, with the exception of ammonium sulphate, the substances which were more readily nitrified were more readily available, the availability being determined by the neutral permanganate method and also by vegetation tests with oats and Hungarian grass.^a This order was dried blood, cotton-seed meal, dried fish, tankage, and finally bone. It was found also that the rate of nitrification was increased by dilution and by the addition of calcium carbonate. The low rate of nitrification of ammonium sulphate was confirmed by a second series of experiments. The following explanation of the low rate of nitrification of ammonium sulphate was offered by us:^b

The low rate of nitrification of ammonium sulphate is probably due to the presence of organisms which nitrify organic compounds in preference to ammonium salts. The presence of the ammonium sulphate may also hinder the activity of the nitrifying organisms. The acids formed may also be a hindrance when no base is present to neutralize them. All three of these causes may be in operation at the same time.

PLAN OF THE EXPERIMENT.

The object of the present experiments was to test the correctness of the above explanation. The experiments upon which we wish to report at this time were conducted upon the same general plan as our former experiments. The sample of soil in each case was sifted through a coarse sieve, and to 500 grams was added an amount of material (ammonium sulphate or cotton-seed meal) containing exactly 0.3 gram of nitrogen. The soil and its contents were thoroughly mixed, transferred to a glass precipitating jar, and placed in a dark closet. Enough water was added from time to time to bring the amount to nearly 15 per cent, the amount which it was necessary to add being determined by weighing one or more of the jars in each set and estimating the loss.

In those cases where calcium carbonate was added the amount which was used was exactly sufficient to combine with the sulphuric acid and the nitrogen of the ammonium sulphate, if the entire amount of the latter were converted to nitric acid.

At the end of three weeks the nitrates were leached out and the amount determined by the Tiemann-Schulze method. The percentages recorded were calculated after deducting the amount of nitrates formed in a blank experiment.

Tests were made for hydrogen sulphid in several cases by placing paper moistened with lead acetate over the jar containing the soil mixture and over the mouth of a flask containing the soil extract acidified and raised to boiling.

SAMPLES OF SOIL USED.

The samples used in the experiment came from localities far removed from each other. They are varied not only as to their natural character, but also in the method of fertilizing, crop grown, etc. They are as follows:

No. 1667.—Pasture soil from the North Carolina station. A light loam containing humus and not acid to litmus.

No. 1668.—Heavy clay soil from the North Carolina station. Contains no humus, possesses low fertility, and is slightly acid to litmus.

No. 1669.—Black garden soil from the Florida station. Contains much humus and is acid to litmus.

No. 1670.—From the Massachusetts Hatch station. Contains little humus and is acid to litmus. This soil is from field C, which has been used in plat experiments for some time, and since 1891 has been fertilized with sulphate of ammonia, muriate of potash, dissolved bone black, and stable manure.

^a Jenkins & Britton, Conn. State Station Report, 1897, p. 357.

^b Bul. No. 176, North Carolina Station, p. 22.

No. 1674.—From the Rhode Island station. Contains humus, is slightly acid to litmus, and has been used in plat experiments.

No. 1675.—A sandy soil with little humus, from the test farm of the North Carolina department of agriculture at Red Springs.

Nos. 1676, 1677, 1678, and 1679 are soils with little humus, from the Rhode Island station. No. 1676 is from plat 23 and No. 1677 from plat 25, both of which have received ammonium sulphate for several years, the former without and the latter with the application of lime. No. 1678 is from plat 27 and No. 1679 from plat 29, both of which have received sodium nitrate for several years, the former without and the latter with the application of lime.

No. 1680.—Sandy soil with little humus, from the test farm of the North Carolina department of agriculture at Tarboro.

The following table shows the amount of nitrates present at the end of three weeks in samples to which no nitrogenous matter was added in the experiment:

Nitrates formed in blank experiments.

No.	Without calcium carbonate.	With calcium carbonate.	No.	Without calcium carbonate.	With calcium carbonate.
	<i>Mg.</i>	<i>Mg.</i>		<i>Mg.</i>	<i>Mg.</i>
1667	10.4	14.0	1676	18.5	(*)
1668	2.5	8.4	1677	14.5	(*)
1669	2.2	(*)	1678	7.3	(*)
1670	4.2	6.5	1679	3.1	(*)
1674	16.3	17.7	1680	1.8	5.0
1675	1.2	(*)			

*Not determined.

The table given below shows the percentages of nitrogen which were nitrified under the conditions stated.

Nitrogen converted to nitrates.

Soil number.	Moisture, average.	Temperature.		Nitrogenous material used.	Nitrified.	
		Extreme.	Average.		Without calcium carbonate.	With calcium carbonate.
	<i>Per cent.</i>	<i>°C.</i>	<i>°C.</i>		<i>Per cent.</i>	<i>Per cent.</i>
1667	10.5	25-30	27	Ammonium sulphate.....	7.2	57.2
				Cotton-seed meal	32.4	43.6
				One-half ammonium sulphate		71.8
				One-half cotton-seed meal.....		43.0
				Ammonium sulphate and cotton-seed meal.....		22.3
1668	13.0	25-30	27	Ammonium sulphate.....	0.6	11.6
				Cotton-seed meal	-0.5	-1.1
				One-half ammonium sulphate.....		43.2
1669	13.0	25-30	27	Ammonium sulphate.....	-0.2	15.8
				Cotton-seed meal	17.3	41.5
				Ammonium sulphate.....	2.2	45.7
1670	15.0	23-28	26	Cotton-seed meal	14.8	32.6
				Ammonium sulphate and cotton-seed meal.....	0.5	
				Ammonium sulphate.....	2.1	2.3
				Cotton-seed meal	3.4	4.9
				One-half ammonium sulphate.....		16.5
1674	10.75	20-26	22	One-half cotton-seed meal.....		10.9
				Ammonium sulphate and cotton-seed meal.....	1.3	

Nitrogen converted to nitrates—Continued.

Soil number.	Moisture, average.	Temperature.		Nitrogenous material used.	Nitrified.	
		Extreme.	Average.		Without calcium carbonate.	With calcium carbonate.
	<i>Per cent.</i>	<i>°C.</i>	<i>°C.</i>		<i>Per cent.</i>	<i>Per cent.</i>
1675	8.0	19-24	20	Ammonium sulphate.....	None.	None.
				Cotton-seed meal.....	None.	None.
1676	12.2	18-23	19	Ammonium sulphate.....	-0.6	1.5
				Cotton-seed meal.....	-1.9	-1.3
				One-half ammonium sulphate.....		1.5
1677	11.7	18-23	19	One-half cotton-seed meal.....		0.6
				Ammonium sulphate.....	1.0	5.6
				Cotton-seed meal.....	1.8	6.2
1678	12.1	18-23	19	Ammonium sulphate.....	-0.3	0.6
				Cotton-seed meal.....	-0.1	-0.3
1679	12.5	18-23	19	Ammonium sulphate.....	1.2	3.0
				Cotton-seed meal.....	2.9	10.6
1680	9.5	19-24	20	Ammonium sulphate.....	-0.1	-1.3
				Cotton-seed meal.....	1.3	-0.8
				One-half ammonium sulphate.....		-2.3
				One-half cotton-seed meal.....		3.2

The following observations are made on the above tables:

(1) Calcium carbonate increases the rate of nitrification of nitrogenous matter already in the soil, and also of ammonium sulphate and of cotton-seed meal added, in those cases where nitrification took place at all.

(2) The rate of nitrification was increased when a smaller percentage of nitrogenous material was added to the soil.

(3) Some soils do not nitrify cotton-seed meal either with or without the aid of calcium carbonate.

(4) Some soils do not nitrify ammonium sulphate either with or without the aid of calcium carbonate.

(5) Some soils which do not nitrify ammonium sulphate nitrify it upon the addition of calcium carbonate.

(6) Hydrogen sulphid is not generated when ammonium sulphate is mixed with soils.

(7) In all soils used by us which nitrify ammonium sulphate and cotton-seed meal, nitrification of cotton-seed meal took place more rapidly than the nitrification of ammonium sulphate in the absence of calcium carbonate.

(8) In soils which nitrify both materials, sometimes in the presence of calcium carbonate, cotton-seed meal is nitrified more rapidly and sometimes less rapidly than ammonium sulphate.

(9) Soils which in their natural state are capable of nitrifying cotton-seed meal lose this power in the presence of ammonium sulphate in case calcium carbonate is absent, and even where calcium carbonate is present one of these materials exercises a retarding action upon the nitrification of the other.

(10) Some soils to which ammonium sulphate and lime have been added for a series of years nitrify ammonium sulphate more rapidly than where there has been no application of ammonium sulphate.

CONCLUSIONS.

From the above observation the following explanations which were previously offered by us (Bul. No. 176, North Carolina station) seem to be sustained:

(1) Ammonium sulphate in some cases hinders the action of nitrifying organisms. In soil No. 1670, 14.8 per cent of nitrogen in cotton-seed meal was nitrified, but upon the addition of ammonium sulphate only 1.3 per cent of the nitrogen was nitrified. In No. 1674 similar results are observed.

(2) In the nitrification of ammonium sulphate, sulphuric acid or nitric acid or both are formed and hinder further nitrification unless neutralized. In soil No. 1668,

7.2 per cent of the nitrogen is nitrified without calcium carbonate, and with calcium carbonate 57.2 per cent. The results in soils Nos. 1669 and 1670 are equally as marked.

(3) Soils differ as to the nitrifying organisms which they contain, some being capable of nitrifying readily both ammonium sulphate and cotton-seed meal; some nitrifying cotton-seed meal more rapidly and some less readily than ammonium sulphate; some nitrifying ammonium sulphate, but not cotton-seed meal, and some being capable of nitrifying either ammonium sulphate or cotton-seed meal.

The following additional conclusions seem justified:

(4) The relative number of organisms in any soil capable of nitrifying ammonium sulphate may be increased by the continued addition of this substance and lime if such germs were originally present.

(5) Calcium carbonate is very helpful in the nitrification of all substances.

The authors wish to express their thanks to Director H. J. Wheeler, of Rhode Island; State Chemist B. W. Kilgore, of North Carolina; Agriculturist W. P. Brooks, of Massachusetts, and Chemist H. K. Miller, of Florida, for their kindness in furnishing samples of soils used in this work.

Mr. FREAR. There is one source of error in the determination of humus or *matière noire* by the official method of which I have seen no mention. As is well known, the method consists in the solution of the humus freed from basic combination by previous extraction with dilute hydrochloric acid in a dilute solution of ammonia. Separation of the ammoniacal solution from insoluble solids is wrought by aid of simple filtration, and from other soluble solids by weighing the total solid residue left upon evaporation and careful drying, igniting it, again weighing the incombustible residue, and estimating the humus as equivalent to the loss by ignition.

The fallacy of the assumption that simple filtration, drying under ordinary conditions in a water-oven, and ignition are sufficient to separate the ammonia-soluble humus from the other soil constituents was very interestingly brought to notice in the course of analyses of certain deep-red Cuban tobacco soils, made under my direction by Mr. C. P. Beistle in my laboratory during the past year.

Mechanical analysis of the ignited soil showed that from 50 to 65 per cent by weight was composed of particles between 0.5 and 0.05 mm in diameter; that the soil, in its present condition of cultivation, behaves like a medium to fine sand. It contains 15 to 18 per cent of particles less than 0.01 mm in size.

The last washings proved, however, to contain much very fine material. The beaker containing the fine silt and clay stood for more than four months undisturbed, and was yet highly discolored with suspended material.

The soil is formed by the weathering of certain chalky formations in the southern part of the province of Habana. Yet the quantity of lime is quite small, 0.27 to 0.37 per cent.

When these soils were treated with dilute ammonia the deflocculating influence of this agent was remarkably exhibited. Nearly the entire body of the soil became finely subdivided, and passed through

single and double filters and single and double hardened S. & S. filters. The ignited residues from the "humus" determination, even when hardened filters were employed, constituted from 60 to 83 per cent of the original weight of air-dry soil. Only upon use of a Pasteur filter were we able to secure a clear solution. The filter was covered with dark, colloidal matter. No data have been accumulated to show the effect of such filtration upon the humus in solution; therefore the results of analysis of the filtrate can not be regarded as trustworthy.

The mere fact that fine particles of mineral matter pass the filter with the humus would be of no consequence if the material were such as to undergo no loss upon ignition; but, in fact, the clay particles which most readily pass the filter are commonly the most highly hydrated mineral materials of the soil. These Cuban soils lost, when ignited, from 17 to 20 per cent of their "water-free" weight. They contained of organic carbon in the surface soils 2.5 to 2.8 per cent, in the subsoil 1.3 to 2.1 per cent. These quantities of carbon correspond to 4 to 5 per cent of humus in the surface soils and to 2.5 to 3.5 per cent in the subsoils. Therefore the principal loss upon ignition was due to elimination of water of combination. The correctness of this conclusion was proven by the determination of water in the combustion products.

The percentages of "humus" extracted by ammonia were, as determined by the official method, from 11.25 to 14.62 per cent of the water-free soil; combustion of several combined residues gave only 1.65 per cent of carbon, corresponding to about 3 per cent of humus. The principal part of the loss by ignition is due, in other words, to the water of combination given up by the hydrated minerals that passed the filter with the humus.

Doubtless the soil upon which this result was obtained is very unusual. Nevertheless, it leaves a serious question as to the value of results obtained by the present method when applied to clay soils. It is desirable that actual determinations of carbon, and possibly hydrogen, be made upon the humus residues as checks upon the analytical work in the case of such soils.

The PRESIDENT. The secretary has some announcements to make before we adjourn.

Mr. WILEY. I have a communication here from the secretary of the Cosmos Club inviting us to meet at the club this afternoon.

(Mr. Wiley at this point read the letter received.)

The PRESIDENT. The Chair will announce the committees provided for by this morning's meeting.

COMMITTEES ON RECOMMENDATIONS OF REFEREES.

A. Phosphoric acid, potash, nitrogen, soils, ash, and insecticides—Messrs. Huston, Ross, Hopkins, Haywood, and Hite.

B. Dairy products, foods, feeding stuffs, and tannin—Messrs. Winton, Penny, Kilgore, Patrick, and Krug.

C. Liquors and food adulterations—Messrs. Bigelow, Caldwell, Withers, Woods, and Munson.

Mr. KILGORE. I would like to offer this resolution:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

The meeting adjourned at 1.30 p. m.

THURSDAY—AFTERNOON SESSION.

President Van Slyke called the meeting to order at 1.25 p. m.

The PRESIDENT. There is an additional paper to be presented on soils, and that will now be in order.

Mr. CAMERON. The paper referred to is entitled "Chemical Examination of Alkali Soils," by Atherton Seidell, and is one of several papers published collectively under the title of "Solution Studies of Salts Occurring in Alkali Soils," as Bulletin 18 of the Division of Soils, United States Department of Agriculture. It will not be necessary to read it at this time, but I move that the paper and the method outlined therein be referred to the committee on recommendations.

The PRESIDENT. The motion has been made that this paper be referred to the committee on recommendations. It is carried.

Is there any business that anyone wishes to bring forward at this time, before we go on with the referee's reports?

Mr. VEITCH. Before we leave the question of soils I have a resolution that I would like to offer. We find the methods very unsatisfactory, and to overcome that condition and get the matter before the association I would like to introduce the following resolution:

Resolved, That the policy of the association as to its way of investigating analytical methods for the determination of available plant food be changed. That, in place of asking the cooperation of all chemists in the comparative study of two or more methods on a furnished set of samples, each of those chemists who are willing to cooperate in the work be asked by the referee to take up such a line of soil study, by any method he may choose, upon known soils of his own State or upon other known samples, the avowed purpose of such a study being the working out of a chemical method for determining the available plant food of soils.

Further, That those who engage in this work shall keep the referee informed of their lines of investigation, shall furnish him with a summary of their results (to be presented by him to the association at the regular meetings), and shall, from time to time as the occasion warrants, present to the association the details of their work.

The PRESIDENT. In my judgment, that resolution would more properly go to the special committee on recommendations of referees, and if there is no objection it will be passed on and submitted to that committee, of which Mr. Huston is chairman.

Mr. HOPKINS. If it is in order, before we leave the subject of soils I would like to make a few remarks regarding the determination of potash by our official method.

SEPARATION OF ALKALIES IN SOIL ANALYSIS BY THE OFFICIAL METHOD.

By C. G. HOPKINS.

In connection with our soil investigations in Illinois, where we are making a large number of analyses, we have had some difficulty in obtaining accurate results by the official method, especially in the separation of the alkalies.

After the precipitation of iron, aluminum, magnesium, etc., by means of barium hydroxid, and of calcium and barium by means of ammonium carbonate, exactly according to the official method (a second precipitation with ammonia and ammonium carbonate having been made), the final filtrate (which is ordinarily simply evaporated to dryness and ignited to secure the weight of the alkaline chlorids) was found to be by no means free from barium.

Even after a third precipitation with ammonia and ammonium carbonate very appreciable amounts of barium remain in the filtrate. Ten separate determinations of the barium remaining in this filtrate (after three precipitations with ammonia and ammonium carbonate) gave the following amounts of barium sulphate:

	Gram.		Gram.
1	0.0065	6	0.0080
2	.0083	7	.0113
3	.0088	8	.0107
4	.0075	9	.0099
5	.0061	10	.0111

It will be seen that this error, in the determination of the alkalies by the official method, is one of considerable importance.

We are now using a modification of the official method in our laboratories, in which we precipitate the last traces of barium by the addition of ammonium sulphate. A sufficient quantity of ammonium sulphate is added so that in the ignition of the residue from the evaporation of the filtrate the alkalies are converted to sulphates and are thus rendered less liable to loss in ignition, an additional advantage possessed by our modification. With the help of Mr. J. H. Pettit, one of our chemists, we are accumulating a considerable amount of data relating to this modified method for separating the alkalies in soil analysis, and now expect to have the matter ready for publication in a few weeks.

I may add that in conversation with others who are familiar with the details of soil analysis I have learned that the official method for the separation of alkalies is commonly regarded as inaccurate, and I suggest that this proposed modification be referred to the proper committee on recommendations.

The PRESIDENT. The paper will be so referred if there is no objection. Is there anything more on soils?

The following letter from Mr. Peter, inclosing analytical data obtained by Mr. Beatty, was then presented:

LEXINGTON, KY., November 7, 1901.

My DEAR SIR: Inclosed herewith please find results on the Association of Official Agricultural Chemists soil samples obtained in this laboratory by Mr. L. O. Beatty, one of our assistant chemists. We did not have time or material to duplicate the results, but Mr. Beatty has been engaged in this kind of work and the figures are

worthy of confidence. The method used in determining the K_2O was somewhat different from that of the referee. An aliquot of the solution corresponding to 100 grams of air-dry soil was evaporated to dryness to separate silica; taken up again with hydrochloric acid and water, filtered, and the solution precipitated with ammonium hydrate and oxalate of ammonia, filtered, and washed. The filtrate and washings were evaporated to dryness, and the ammonia salts expelled by careful heating. The potassium was then determined in the usual way by platinum chlorid.

Very respectfully,

ALFRED M. PETER.

Mr. F. P. VEITCH,

Associate Referee, Washington, D. C.

Results of work by Mr. L. O. Beatty, Kentucky Agricultural Experiment Station, on the soil samples for Association of Official Agricultural Chemists.

Laboratory number	8594.	8595.	8596.	8597.	8598.
Referee's number	1.	2.	3.	4.	5.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Moisture, drying in water-oven	1.60	1.04	1.89	0.93	1.09
Combined water, organic matter, etc	6.48	2.77	7.11	4.88	5.40
Humus, official method	1.81	.32	2.56	1.55	1.98
K_2O by N/5 HCl, 5 hours' digestion at 40° C.; precipitated out the iron, alumina, and lime with ammonia and ammonium oxalate, and expelled ammonium salts by heat	.1194	.0215	.0152	.0123	.0168
K_2O same, except the temperature of digestion reached 50°	.1120				
K_2O by N/5 HNO_3 , digested 5 hours at the room temperature (about 24°); iron, etc., separated as above	.0838	.0181	.0133	.0106	.0127
P_2O_5 by N/5 HCl, digested 5 hours at 40° C.; determined volumetrically	.1760	.0686	.1223	.0465	.0215
P_2O_5 by N/5 HNO_3 , digested 5 hours at room temperature; determined volumetrically	.1688	.0650	.1088	.0275	.0134

The PRESIDENT. Next in order is the report on ash. The referee is not present. Is the referee on foods and feeding stuffs ready to report?

REPORT ON CATTLE FEEDS.

By WILLIAM H. KRUG, *Referee*.

A circular letter requesting cooperation in the work on feeding stuffs brought favorable replies from 16 chemists, to whom the following samples were sent:

No. 1. Wheat.

No. 2. Bran.

No. 3. Clover seed.

The suggestions embodied in the report for 1900 were made the basis for the following recommendations as to lines of research which appeared worthy of attention:

I. *Moisture*.—Determine the moisture in all samples by the following methods:

a. Twenty-four hours in a water bath at a temperature at which water boils in your locality. State this temperature in your report.

b. To constant weight in a water bath as in a.

c. To constant weight in an air bath at 100° C., or if constant weight can not be obtained, until two consecutive dryings of an hour each show a loss of less than 2 mg. State the method pursued.

d. As prescribed in Bulletin 46, revised edition (U. S. Department of Agriculture, Division of Chemistry), page 23, 11, 2.

e. To constant weight as in *d.*

f. To constant weight in vacuo at 70° C.

Starch—

a. Determine the starch in samples 1 and 2.

b. Collect the cuprous oxide on a tared Gooch crucible and weigh as such, calculating it as Cu_2O .

c. Determine the solvent action of the diastase and water on the pentosan by evaporating an aliquot part of the solution to a small volume, and distilling with 12 per cent hydrochloric acid.

Pentosan—

a. Determine the pentosan in samples 1 and 2.

b. Determine the effect which variations in the time of standing after precipitation with phloroglucol have on the quantity of phloroglucid obtained. Make a series of determinations in which the precipitated solution is allowed to stand at least twenty hours.

c. Determine the effect of the rapidity of distillation on the quantity of furfural obtained by regulating the burner so as to distill over 20, 25, 30, etc., cc per ten minutes.

Galactan.—Determine the galactan in sample 3.

There has been an evident lack of interest in the work this year, and only four reports have been received.

THE ESTIMATION OF MOISTURE.

The results reported on last year's samples showed such wide and startling variations that it appeared desirable to investigate the causes which led to such errors in a determination apparently simple and easy of execution. Various methods of drying were suggested, and the results are given in the following table:

TABLE I.—*Moisture determinations.*

Analyst.	Method of drying.	Sample No. 1.	Sample No. 2.	Sample No. 3.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
G. S. Fraps, North Carolina	Caldwell water oven and Caldwell tubes, 7 hours.	11.81	11.34	
F. S. Shiver, South Carolina	Water oven, 24 hours.....	11.15	10.56	6.99
T. M. Price, Maryland	do	10.38	10.58	6.67
F. S. Shiver, South Carolina	Water oven, constant weight, 99° C...	11.15	10.54	7.31
E. B. Holland, Massachusetts	Glycerin and water oven, about 100° C., 29 hours.	11.06	10.05	7.07
W. H. Krug, U. S. Department of Agriculture.	Water oven, 98° C., 26 hours		9.84	
J. H. Norton, U. S. Department of Agriculture.	In vacuo, 70° C., 10 hours		9.56	
W. H. Krug, U. S. Department of Agriculture.	In vacuo, 70° C., 15 hours	10.84	10.19	6.65
J. H. Norton, U. S. Department of Agriculture.	Air bath, 100° C., 7 hours.....		9.96	
W. H. Krug, U. S. Department of Agriculture.	Air bath, 100° C., 5 hours.....		10.14	
Mean of all determinations		11.06	10.28	6.94

Mr. Fraps reports that when the samples were dried five hours in a Caldwell oven, using Caldwell tubes, the following results were obtained: No. 1, 11.75 per cent; No. 2, 11.07 per cent. Two hours additional gave the results found in the table, showing that there was only a slight loss in weight after five hours.

The figures presented this year show a marked improvement when compared with those reported in 1900, but differences still exist, and these are most marked when we compare the results obtained in vacuo at 70° C. with those obtained by drying in air, the former being almost uniformly lower, although the drying was continued until no further loss occurred, the time required to reach this point being in every case fifteen hours. It is evident that where materials of this kind are dried in air, this being the method most generally followed, the final result does not represent the absolute moisture present, but is increased by losses due to exposure to the air at the temperature employed. The official methods for the analysis of foods direct that the material shall be dried for five hours, at the temperature of boiling water, in a current of dry hydrogen, or in vacuo. Neither of these methods has been generally adopted, as most laboratories are not equipped with the necessary facilities and are thus forced to use some other method. In such a case the ordinary air bath should, however, never be used, as it is practically impossible to control the temperature, even on the same shelf, whereas this is easily accomplished when a water oven is employed. It being generally acknowledged that the losses noted when a material is dried in air are not wholly due to moisture, it is naturally not feasible to carry the drying to the point where a gain in weight takes place, as this requires considerable time and is of no advantage, since it does not yield an absolute value. A time limit must therefore be fixed, and in view of the fact that in most cases the moisture determinations are made by drying the substance in air, it seems advisable to incorporate certain directions in the official methods, and your referee would suggest that we adopt, as optional in the case of feeding stuffs, the method given for sugars, which is as follows:

Dry from 2 to 5 grams in a flat dish (nickel, platinum, or aluminum), at the temperature of boiling water, for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there be only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not great.

THE ESTIMATION OF STARCH.

So little work has been done on the diastase method this year that it is impossible to draw conclusions or to make any suggestions as to changes. The results are given in Table II. Owing to the different methods used to weigh the copper, the results are not averaged.

TABLE II.—*Starch determinations.*

Analyst.	Sample 1.		Sample 2.	
	In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
F. S. Shiver, South Carolina	^a 53.68	60.42	^a 8.57	9.58
J. H. Norton, U. S. Department of Agriculture.....	^b 56.55	63.43	^b 13.95	15.53
Do.....	^c 56.03	62.84	^c 13.50	15.03

^a Copper weighed as CuO.

^b Copper weighed as Cu₂O.

^c Copper weighed as Cu.

The figures reported by Mr. Norton represent in each case an average of six determinations, and the amount of copper obtained by directly weighing the oxid and assuming its composition to be Cu₂O was in every instance higher than when the oxid was dissolved and the copper obtained electrolytically, showing thereby that

the red oxid is not uniform in composition and affords but an uncertain basis for calculation. This is in accord with the statements made in the literature from time to time by European chemists.

THE ESTIMATION OF PENTOSAN.

The results are given in the following tables:

TABLE III.—*Pentosan determinations.*

Analyst.	Sample 1.		Sample 2.	
	In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
F. S. Shiver, South Carolina.....	7.57	8.52	25.12	28.08
E. B. Holland, Massachusetts.....	7.08	7.96	24.71	27.47
G. S. Fraps, North Carolina.....	6.30	7.14	23.00	25.97
T. M. Price, Maryland.....	7.83	8.74	26.01	29.09
J. H. Norton, U. S. Department of Agriculture.....	6.76	7.58	25.20	28.06
Average.....	7.11	7.99	24.81	27.73

COMMENTS.

G. S. Fraps.—It is impossible to dry the phloroglucid precipitate to constant weight by heating three to four hours, especially if it is large. The following figures were obtained by drying four hours, and continuing the drying:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>	<i>Grams.</i>
Weight, 4 hours.....	0.2135	0.2227	0.8932	1.1741	1.0786
Weight, 6 hours.....	.2037	.2127	.7194	.7443	.6621
Loss.....	.0098	.0100	.1738	.4298	.4165
Eight hours.....					.6563
Loss.....					.0058

It would be easy to give a number of examples. I usually dry six hours, weigh, and then dry two more hours.

A determination of furaloid^a was made in the distillates by redistilling them. The crude furfural from sample No. 1 contained 19 per cent, No. 2, 12 per cent furaloid.

Mr. Holland reported in detail the individual determinations, showing a very close agreement. He determined the pentosan in both samples, with and without previous extraction with ether, and obtained the following figures: No. 1, not extracted, 7.18 per cent; extracted, 6.98 per cent; No. 2, not extracted, 24.69 per cent; extracted, 24.73 per cent. These data indicate that previous extraction with ether is not necessary, except, possibly, in the case of substances containing a very high percentage of fat.

In the directions sent out with the samples your referee suggested that experiments be made to determine the effect which the time of standing after precipitation has

^a See Am. Chem. Jour., 1901, 25, 502.

on the yield of pentosan. Considerable work has been done on this point, and the results are given in the following table:

TABLE IV.—*Influence of time precipitate is allowed to stand on yield of pentosan.*

Analyst.	Time of distillation per 30 cc.	Time precipitate was allowed to stand.	Sample No. 1.		Sample No. 2.	
			In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Minutes.	Hours.	Per cent.	Per cent.	Per cent.	Per cent.
T. M. Price, Maryland	10	1	7.83	8.74		
Do.	10	5	7.83	8.74		
J. H. Norton, U. S. Department of Agriculture	10	16			24.90	27.73
Do.	10	20	6.76	7.58	25.20	28.06
F. S. Shiver, South Carolina	10	20	7.57	8.52	25.12	28.08
G. S. Fraps, North Carolina	10	20	6.30	7.14	23.00	25.97
E. B. Holland, Massachusetts	9.5	20+	7.18	8.07		
Do.	10+	20+			24.69	27.45
Do.	10	20+			24.73	27.49
Average			6.95	7.83	24.55	27.41
E. B. Holland, Massachusetts	10.25	21	6.98	7.85		
J. H. Norton, U. S. Department of Agriculture	10	24			25.68	28.59
T. M. Price, Maryland	10	24	7.83	8.74	26.01	29.09
Average					25.85	28.84
T. M. Price, Maryland	10	48	7.83	8.74		

The considerable variations which exist between the individual data obtained by a supposedly uniform method of working render it rather difficult to base any definite conclusions on these results. They can only be accepted as an indication that an increase in the time of standing from twenty hours (as now directed) to twenty-four hours results in a higher yield of phloroglucid. Further confirmation is, however, necessary before any such change in the present method is warranted. In connection with this point we must, furthermore, consider the fact discovered by Mr. Fraps, that the distillate always contains a substance which separates as a black precipitate on standing, and one or more substances which are not furfural, but are precipitated by phloroglucol. He has given the latter the name "furaloid," and has published an interesting paper on the subject in the American Chemical Journal, volume 25, No. 6, page 501. His summary is as follows:

The distillate from vegetable materials with hydrochloric acid may contain, besides other things, fatty acids, a substance which separates as a black precipitate if the vessel is allowed to stand, and a substance (or substances) which is destroyed by distilling again (furaloid). All three of these may appear in the phloroglucol precipitate.

The furaloid-yielding bodies were found in all the materials treated, the crude furfural in the hydrochloric acid distillate containing from 7 to 23 per cent of furaloid.

The furaloid-yielding bodies are hydrolyzed by boiling 1.25 per cent sulphuric acid, and lost in the subsequent evaporation. They are digested to a greater extent than the total pentosans in the one sample of timothy hay excrement tested.

The furaloid is not formed by the action of the hydrochloric acid on the pentose sugars.

While there are thus several sources of error in the present method, in so far as its value as a means of determining the actual amount of pentosans present is concerned, Mr. Fraps does not believe it expedient to vary the method so as to permit the

determination of the furfural and furaloid separately, as they can be very conveniently grouped together. An experiment made by him showed that the furaloid-yielding bodies are somewhat more digestible than the pentosan.

Your referee further suggested that experiments be made to determine the influence of the time of distillation on the yield of pentosan. The results are given in Table V.

TABLE V.—*Influence of time of distillation on yield of pentosan.*

Analyst.	Time of distillation per 30 cc.	Time precipitate was allowed to stand.	Sample No. 1.		Sample No. 2.	
			In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Minutes.	Hours.	Per cent.	Per cent.	Per cent.	Per cent.
J. H. Norton, U.S. Department of Agriculture	5	20			21.33	23.75
Do.....	10	20	6.76	7.58	25.20	28.06
E. B. Holland, Massachusetts	9.5	20+	7.18	8.07		
Do.....	10.25	21	6.98	7.85		
Do.....	10+	20+			24.69	27.45
Do.....	10	20+			24.73	27.49
Average			6.98	7.83	24.87	27.67
J. H. Norton, U.S. Department of Agriculture	15	20			24.84	27.66
E. B. Holland, Massachusetts	15	22+	6.83	7.68		
Do.....	15	20+			22.56	25.08
T. M. Price, Maryland.....	15	24			24.80	27.73
Average					24.07	26.82

It is very evident that the time of distillation at present prescribed by this method is best, as it gives the highest results. The primary object of the investigation was to determine if the time of distillation could be shortened, as this feature of the method is tedious and time-consuming. Mr. Norton's results in a series of determinations showed conclusively that when the time of distillation was reduced to five minutes for every 30 cc the yield of pentosan was decreased as much as 4 per cent. Neither does an increase of the time to fifteen minutes possess any advantages, the results indicating that this also causes a slight decrease in the yield.

E. Kröber^a has recently published a long and exhaustive study of the phloroglucol method, and his conclusions may be briefly summarized as follows:

- (1) That the result is not influenced by the length of time the precipitates stand.
- (2) That the precipitate is best collected in a gooch.
- (3) That the wash water must be added in small portions and always before the precipitate in the gooch has become too dry.
- (4) That the precipitate must not contain hydrochloric acid, as the presence of the acid leads to high results, owing to oxidation.
- (5) That the presence of diresorcol in the phloroglucol does not influence the result.
- (6) That about twice as much phloroglucol must be added as there is furfural present.
- (7) That the phloroglucol must previously be dissolved in dilute hydrochloric acid (sp. gr. = 1.06).

^a Journal für Landwirtschaft, 48, 537.

(8) That a uniform time of drying is indicated (four hours at 98.5° to 100° C.), as the last traces of water are very difficult to remove and eventual oxidation will cause errors.

(9) That the gooch must be weighed in a weighing bottle, as the phloroglucid is very hygroscopic.

The author further recommends that the quantity of substance taken for distillation be such that the weight of the phloroglucid shall not exceed 0.300 gram.

The chief difference between these conclusions and the results obtained by the chemists of this association is found in the statement that the presence of diresorcol does not affect the final result. In support of this the author gives a series of results obtained by precipitating a solution of furfural with pure and impure phloroglucol. The averages are as follows:

	Weight of phloroglucid.	
	Series 1.	Series 2.
	<i>Gram.</i>	<i>Gram.</i>
Pure phloroglucol.....	0.08755	0.04035
Impure phloroglucol.....	.08760	.04055

This point is, however, not of much importance, as the official method prescribes a convenient process for the purification of the phloroglucol. The suggestions made by Kröber with reference to the precautions to be taken in drying and weighing are important, and your referee believes it advisable to incorporate them in our method.

THE ESTIMATION OF GALACTAN.

The results obtained are given in the following table.

TABLE VI.—*Galactan determinations.*

Analyst.	Sample 3.	
	In original sample.	Calculated to water-free basis.
	<i>Per cent.</i>	<i>Per cent.</i>
F. S. Shiver, South Carolina.....	5.12	5.52
E. B. Holland, Massachusetts.....	5.05	5.43
T. M. Price, Maryland.....	3.07	3.29
J. H. Norton, U. S. Department of Agriculture.....	5.73	6.14
Average.....	4.74	5.09

With the exception of the figures reported by Mr. Price, these results are satisfactory. The conversion of galactan into mucic acid is never complete in practice and it is therefore impossible to obtain the theoretical yield. This fact in itself is such a possible source of error that very closely agreeing results can not be expected.

RECOMMENDATIONS.

The recommendations this year deal mainly with minor changes tending toward more exact methods.

Moisture.—It is recommended that the method used for the drying of sugars be adopted as optional for the drying of feeding stuffs.

Phloroglucol method.—It is recommended that—

a. The first line be changed so as to read, "A quantity of the material, chosen so that the weight of the phloroglucid obtained shall not exceed 0.300 gram, is placed in a flask." The use of 3 grams of material, as now specified, often leads to such large quantities of phloroglucid that filtration, subsequent washing, and drying of the precipitate are very difficult. By basing the quantity of material on a definite weight of phloroglucid which is small enough to present no difficulties, this will be avoided.

b. In the seventh line the words "by means of a separatory funnel and" be inserted after "added." This insertion is merely explanatory, as there appears to have been some doubt in the past as to the form of apparatus which should be used.

c. In the seventeenth line the words "from three to" be stricken out. Your referee believes that the fixation of a time limit in connection with the first recommendation will tend to give more concordant results.

d. In the eighteenth line the words "in a weighing bottle" be inserted after "weighed." The hygroscopic nature of the phloroglucid renders the necessity of this precaution self-evident.

Galactan method.—It is recommended that—

a. In the twelfth line the word "gently" be stricken out, the words "at 80° C" be inserted after "bath," and the words "with constant stirring" after "minutes." The directions are rather indefinite at this point, and it was thought advisable to fix a definite temperature of digestion. In the absence of definite information, the temperature used in the laboratory of the Hatch Experiment Station was chosen. The object of the digestion with the ammonium carbonate solution is to convert the mucic acid into ammonium mucate, and this is naturally facilitated by stirring the mixture constantly.

b. In the seventeenth line the words "avoiding unnecessary heating, which causes decomposition," be inserted after "bath." This insertion is a necessary precaution, as the residue obtained on evaporation of the filtrate rapidly darkens when subjected to further heating.

c. In the twenty-first line the words "three hours" be substituted for "a short time." A definite time of drying appears preferable to the directions now given.

The PRESIDENT. Any paper relating to this subject is now in order.

THE DETERMINATION OF PENTOSAN-FREE CRUDE FIBER.

By G. S. FRAPS.

Until a few years ago, the only demand made upon the method of determining crude fiber was that the crude fiber should not contain appreciable quantities of nitrogenous bodies. A method was chosen which would give a crude fiber having a minimum nitrogen content, always bearing in mind other considerations, as its convenience, accuracy, and ability to give concordant results in the hands of different analysts.

In the last few years a determination has been introduced into our scheme of analysis for foods which compels us to demand more of the crude fiber determination. The introduction of the pentosan determination compels us to choose between two courses—to require that the crude fiber should not contain appreciable quantities of pentosans, or to make two determinations of pentosans when necessary—one of total pentosans and one of pentosans in crude fiber.

Does the crude fiber prepared by the official method contain pentosans? This question has been answered in the affirmative. The crude fiber contains pentosans,

often in considerable quantity, as is shown by the following figures selected from the large number available:

TABLE I.—*Pentosans in 100 parts of crude fiber.*

J. König, Analyst, 23 , 47 (1898):	Per cent.
Rye straw	18.4
Pea straw	15.0
Clover hay	12.7
Wheat bran	2.6
Bulletin 172, N. C. Station (1900):	
Timothy hay	14.4
Crabgrass hay	13.4
Green rape	7.7
Rice bran	8.3

Since the official method does not yield a pentosan-free crude fiber, we have the choice of two alternatives: To adhere to this method and determine pentosans in crude fiber when necessary, or to seek for a new method by which a pentosan-free crude fiber may be prepared.

KÖNIG'S METHOD.

In an article read before this body last year the writer directed attention to the method of J. König for determining pentosan-free crude fiber. This consists in boiling the material with glycerol containing sulphuric acid, the boiling point of the glycerol being 131° to 133° C.^a

Kellner, Herring, and Zähner tested this method, and decided that it is an improvement over the one in present use, both in speed and accuracy.^b

C. Beck^c did not decide quite so favorably. He found it difficult to keep the temperature of the glycerol between 131° and 133°, and consequently obtained results which varied somewhat. He concluded that the method may be particularly useful for fodders, but for finely ground grain feeds the other method is more reliable.

König replied that the method was better adapted for grain feeds. The temperature could easily be kept at 131° to 133° if glycerol of exactly 1.229 specific gravity is used, and the boiling conducted so gently that only a few drops of water collect in the condenser.

During the past summer the writer undertook some study of König's method. The full description of the method, with such precautions and observations as seem necessary, is as follows:

DETERMINATION OF CRUDE FIBER.

(a) *Glycerol-sulphuric acid.*—Determine the specific gravity of the glycerol by means of a picnometer or specific-gravity balance. A hydrometer can not be used, as on glycerol its readings would not be correct. The per cent of glycerol may be calculated by the following table:

Determination of glycerol by specific gravity.

Specific gravity at 15.5°.	Glycerol.	Specific gravity at 15.5°.	Glycerol.	Specific gravity at 15.5°.	Glycerol.
	<i>Per cent.</i>		<i>Per cent.</i>		<i>Per cent.</i>
1.2674	100	1.2540	95	1.2406	90
1.2647	99	1.2513	94	1.2380	89
1.2620	98	1.2486	93	1.2353	88
1.2594	97	1.2460	92	1.2327	87
1.2567	96	1.2433	91	1.2300	86

^a Analyst, **23**, 47, or Exp. Station Record, **9**, 1021.

^b Exp. Station Record, **11**, 705. ^c Ibid., **12**, 611.

Should the temperature of the specific-gravity determination not be made at 15.5°, a correction may be made by adding 0.00058 for each degree above that temperature.

Dilute the glycerol to 86 per cent, or 1.230 specific gravity, and make up a solution containing 20 cc sulphuric acid (1.84 specific gravity) per liter of the glycerol-acid mixture.

(b) *The determination.*—Place 3 grams of substance in a 500 cc Erlenmeyer flask, add 200 cc glycerol-sulphuric acid, and connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Heat to boiling, and boil very gently for an hour, shaking the flask from time to time, so as to wash down any particles. The boiling should take place in such a manner that only a few drops of water are condensed. Prepare a thin layer of asbestos in a 2-inch Hirsch funnel, and on this place a perforated platinum disk. Filter the glycerol on this, using a suction pump. The mixture should be shaken well before it is poured on the filter, and each flask should have the full benefit of the suction pump when the filtration starts, until the glycerol begins to come through. Wash with hot water, with alcohol, and with a mixture of equal volumes of alcohol and ether. The alcohol and ether remove, besides fats, certain products not soluble in water, formed by the action of the glycerol acid. Transfer to a platinum dish, dry, weigh, and incinerate completely. The loss in weight is crude fiber.

NECESSARY PRECAUTIONS.

A primary necessity is to have the glycerol of the required density. A comparatively slight variation in density will cause an increase or decrease in boiling point, with a corresponding decrease or increase in the crude fiber.

On account of the nature of glycerol, it is necessary to filter it as hot as possible and before it cools. Cold glycerol, even if diluted with water, filters very slowly indeed. The following method of filtration is recommended as the best, after trying several ways:

(1) Dilution with water and filtration while hot, as recommended by König. It seems impossible to get the liquid through the filter sometimes when this method is used. It always takes longer than the method recommended, if properly carried out.

(2) Dilution with alcohol. Filtration takes place rapidly, and this method may be used in difficult cases. It is possible that the alcohol may precipitate some of the products of the action of the acid on the material.

RESULTS.

The method is much shorter than the official method. The writer has weighed out a set of samples, digested, filtered, and washed the crude fiber in three hours. Concordant results are more easily obtained than by the other method. Some of the determinations made are as follows:

TABLE II—*Determination of crude fiber by König's method.*

	Per cent.		Per cent.
Timothy hay.....	{ 27.40	Timothy hay, No. 2	{ 31.95
	{ 27.65		{ 31.60
	{ 27.32		{ 32.04
Corn bran.....	{ 10.09	Sheep dung	{ 34.33
	{ 9.80		{ 33.94
	{ 10.19		
	{ 10.41		

The work of König and others has shown that the crude fiber is practically free from pentosans.

In the case of cotton-seed meal great difficulty was experienced in filtration, and the crude fiber obtained was much too high. It seems probable that this substance must be subjected to some preliminary treatment before the method can be applied.

This method was given to a graduate student, with the request that he determine crude fiber in two samples. His determinations were 3.5 to 4.9 per cent lower than those of the writer, and the explanation lies in the fact that the glycerol he used was a little more concentrated, and he boiled the mixture more vigorously.

It is believed that determinations made by different analysts with the König method will compare favorably with those made by the official method, especially when chemists have become to some extent familiar with the new method.

CONCLUSION.

The König method has the following advantages:

- (1) It yields a fiber practically free from pentosans.
- (2) It requires fewer manipulations and less time than the official method.

On the other hand, it requires to be studied in its application to cotton-seed meal.

The writer desires respectfully to suggest to the referee on foods that the study of the König method of determining crude fiber be undertaken.

The PRESIDENT. If there is no objection this subject will be referred to committee B on recommendations, of which Mr. Winton is chairman. Is there anything further in relation to the analyses of foods and feeding stuffs? If not we will listen to the report on ash.

REPORT ON ASH.

By G. S. FRAPS, *Referee*.

The referee of the present year continued the work on ash along the line adopted by Mr. Shuttleworth, the referee last year, namely, the study of the method of preparing the ash. The following circular letter was sent out to the chemists of all the agricultural experiment stations in the country:

DEAR SIR: The referee on ash of the Association of Official Agricultural Chemists this year will continue the study of the method of preparing the ash. The preparation of an ash that contains all the inorganic constituents lies at the foundation of the ash analysis; it would be worse than useless to analyze an ash from which unknown amounts of chlorin, sulphur, potash, soda, etc., have been volatilized. The point to which special attention will be given this year is, whether any loss of sulphur or potash occurs when the substance is incinerated with calcium acetate in an open dish.

We would like to have your cooperation in this work. Please inform the referee if you will undertake it.

Yours, very truly,

G. S. FRAPS, *Referee*.

E. W. MAGRUDER, *Associate Referee*.

Sixteen chemists requested samples and undertook to perform the work if time permitted. Encouraging letters were received from others who were unable to cooperate. W. P. Headden called attention to some work of his relative to loss in sulphur and chlorin in preparing an ash.^a

Two samples were prepared—of wheat bran and cotton-seed meal. Calcium acetate was prepared by dissolving chemically pure calcium carbonate in chemically pure acetic acid and evaporating the solution to dryness. The following directions for work were sent along with the samples:

DIRECTIONS FOR WORK.

Mix each sample thoroughly and determine moisture in the usual way. Determine potash and sulphur in duplicate in both samples by the methods described below.

Calcium acetate.—Dissolve the contents of the bottle in water and dilute to 200 cc in a graduated flask; 100 cc will contain calcium acetate approximately equal to 1 gram CaO.

^a Colorado Experiment Station. Bul. No. 35, 1896.

Sulphur and potash.—Place 15 grams material in a weighed platinum dish and moisten it with 40 cc of the calcium acetate solution. Mix well, dry on a water bath, and ignite to an ash at as low a temperature as possible. Weigh. Dissolve the ash in dilute hydrochloric acid, evaporate to dryness and dry the residue with a little hydrochloric acid, take up with about 50 cc of boiling water, filter, and wash and determine the sulphur by the official method, page 78, Methods Association of Official Agricultural Chemists (1899).

Heat the filtrate, add sulphuric acid to precipitate the barium, and add ammonia and ammonium oxalate. Filter, wash with hot water, and determine potash in the entire filtrate as directed in (3a), page 22, Methods Association of Official Agricultural Chemists.

Potash.—Ignite 15 grams material with sulphuric acid as directed in (b), page 22. Add ammonia and ammonium oxalate to the hot solution, filter, wash with hot water, and proceed to determine potash in the entire filtrate as directed in (3a), page 22.

Please report results as soon as possible, not later than October 1. State weight of material taken, weight of ash obtained by ignition with calcium acetate, weight of precipitates obtained, and percentage of K_2O and SO_3 in the wheat bran and cotton-seed meal.

MOISTURE.

The results on moisture are stated as a matter of record. The fact that moisture determinations may vary considerably has already been emphasized by the referee on foods.

TABLE I.—*Moisture determinations.*

Analyst.	Wheat bran.	Cotton-seed meal.
	<i>Per cent.</i>	<i>Per cent.</i>
C. H. Jones, Vermont	8.93	4.82
E. G. Runyan, Washington, D. C.	9.16	4.82
T. M. Price, Maryland *	10.18	5.48
F. S. Shiver, South Carolina ^b	10.43	5.62
R. W. Thatcher, Washington (State) ^c	11.18	6.42

* Dried sample to constant weight at 100° C.

^b Dried sample to constant weight in water oven.

^c Dried sample in hydrogen at the temperature of boiling water.

ASH.

The quantity of ash obtained from 15 grams of material, as received, is given in the following table. The ash contains calcium oxid and carbonate derived from the calcium acetate. Its quantity gives a clue as to the temperature used in ignition.

TABLE II.—*Ash.*

Analyst.	Wheat bran.	Cotton-seed meal.
	<i>Grams.</i>	<i>Grams.</i>
C. H. Jones, Vermont	1.1754	1.5036
E. G. Runyan, Washington, D. C.	1.2503	1.5377
T. M. Price, Maryland	1.1754	1.4729
T. S. Shiver, South Carolina	1.2817	1.6172
R. W. Thatcher, Washington (State)	1.2243	1.5100
A. W. Blair, Florida	1.1828	1.4826

REMARKS OF ANALYSTS.

C. H. Jones.—The weight of ash reported contains the added lime and considerable carbon which I could not get rid of by heating.

T. S. Shiver.—These determinations of ash may be a little high, since a small quantity of carbon, especially in the case of the cotton-seed meal, was still left in the same. I have never found simple ignition satisfactory in the determination of ash, and therefore it is my invariable practice to first char the sample, and then extract with hot water. The aqueous extract is evaporated to dryness, gently ignited, and weighed. The insoluble residue is ignited to redness until all carbon is destroyed, and also weighed. By these means I get an ash entirely free from carbon, and also avoid any loss of volatile constituents. In the weights of ash given for the cotton-seed meal and the wheat bran, I have not made corrections for the amount of lime contained in the calcium acetate used.

R. W. Thatcher.—Forty cubic centimeters of calcium acetate solution ignited alone gave 0.6782 gram residue. Correcting for this addition the percentage of ash is:

	Per cent.
Cotton-seed meal	5.51
Wheat bran.....	3.64

E. G. Runyan.—Ash was determined in the usual way, using 3 grams:

	Per cent.
Cotton-seed meal	6.87
Wheat bran.....	4.75

The remainder of my report falls naturally into two sections; namely, the determination of sulphur and the determination of potash.

DETERMINATION OF SULPHUR.

The results obtained for sulphur according to the methods already described are given in the following table.

TABLE III.—*Sulphur (SO_3) by calcium acetate method.*

Analyst.	Bran.		Cotton-seed meal.	
	Grams taken.	Per cent.	Grams taken.	Per cent.
C. H. Jones, Vermont	3.7500	0.19	3.7500	0.60
E. G. Runyan, Washington, D. C.	1.5000	.18	1.5000	.30
T. M. Price, Maryland	6.0000	.18	6.0000	.36
T. S. Shiver, South Carolina	15.0023	.21	15.0061	.39
R. W. Thatcher, Washington (State) ..	15.0000	.19	15.0760	.37
G. S. Fraps, North Carolina	15.0000	.22	15.0000	.34
A. W. Blair, Florida.....	15.0000	.18	15.0000	.36

Mr. E. G. Runyan gives determinations of sulphur in the ash prepared in the usual way, as follows: Wheat bran, 0.015 per cent SO_3 ; cotton-seed meal, 0.026 per cent. Ignition with calcium acetate therefore gives over ten times as much sulphur in each case as when the material is ignited alone.

VALUE OF THE METHOD.

The results obtained by different analysts with the calcium acetate method agree to a certain extent, and from this standpoint the method could be considered as fairly satisfactory.

Study of the method in another way, however, warrants the assertion that the calcium acetate is valueless so far as the determination of sulphur is concerned. This conclusion is reached after comparing the results obtained by the calcium acetate method with those obtained by the use of another method.

A method was sought by which we could be reasonably certain that all the sul-

phur contained in plant materials, alone or in organic combinations, would be retained and determined. A great difficulty is that a very small amount of sulphur is contained in a large quantity of plant materials. Several methods were given a trial and finally the one described below was adopted.

NITRIC-ACID METHOD FOR SULPHUR.

Place 15 grams of material in a flat porcelain evaporating dish of about 250 cc capacity, add 35 cc of nitric acid (concentrated), and heat gently until the action has moderated; add 1 gram potassium nitrate and evaporate to a thin paste. Transfer to a platinum dish, evaporate on a water bath, and ignite to an ash. Heat for some time, remove to a porcelain dish and dissolve the ash in dilute hydrochloric acid, evaporate to dryness, and dry the residue thoroughly to render silica insoluble. Moisten with a little dilute hydrochloric acid, evaporate, and dry again. Moisten the residue with about 5 cc of hydrochloric acid, take up with about 50 cc of boiling water, filter, and wash, and determine the sulphur by the official method. (Methods Association of Official Agricultural Chemists, 1899, page 78.)

Make a blank determination, using the same quantities of potassium nitrate and nitric acid, and evaporating off the acid before precipitating.

This method was sent out to a few analysts. The results obtained are as follows:

TABLE IV.—*Determination of Sulphur as SO₃.*

WHEAT BRAN.

Analyst.	In ash.	In calcium acetate ash.	Nitric-acid method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. H. Jones, Vermont		0.191	0.311
A. W. Blair, Florida		.173	.396
Do.....		.182	.393
G. S. Fraps, North Carolina		.221	.398
Do.....			.336
Do.....			.368
Do.....			.373
Do.....			.369
Average		.194	.368

COTTON-SEED MEAL.

C. H. Jones, Vermont		0.597	0.964
A. W. Blair, Florida.....		.366	.965
Do.....		.342	.981
Do.....		.367	*1.09
G. S. Fraps, North Carolina		.343	.928
Do.....		.328	.930
Do.....			.941
Average		.390	.951

VARIOUS FEEDING STUFFS.

Green rape (G. S. Fraps)	1.02	1.25	1.63
Wheat bran (G. S. Fraps)	.00	.14	.30
Corn silage (G. S. Fraps)	.20	.24	.43
Timothy hay (G. S. Fraps)	.15	.17	.28

* This was the first one worked by this method and was not satisfactory. It was excluded from the average.

CONCLUSIONS.

In considering these figures it must be remembered that 15 grams of material is used, and on this account the sulphur trioxid can be calculated to 0.001 per cent.

In the light of these results the only conclusion in regard to the calcium acetate method is that it does not give correct values for sulphur. It is not believed that the loss of sulphur is due to volatilization of sulphates, but to the escape of organic sulphur compounds, which are not burned or oxidized. Most of the sulphur in a plant is not in the form of sulphates, but in an organic form, and it is not surprising that this is lost under such conditions. The same would be true of chlorin, which is probably much more difficult to retain than sulphur.

The nitric acid method gives fairly satisfactory results in the hands of different analysts, and taken in connection with the following work these results, in my opinion, justify its adoption by the association.

MODIFICATION OF THE NITRIC ACID METHOD.

The nitric acid method has been modified in two ways as follows:

(1) Five grams of material was placed in a $3\frac{1}{2}$ -inch porcelain evaporating dish, 20 cc of nitric acid (concentrated) added, and the mixture heated cautiously on a water bath until all danger of overflowing had passed. It was then evaporated partly, 10 cc of a 5 per cent solution of potassium nitrate added, the mixture evaporated to complete dryness, and ignited, at first gently, then under a blast lamp, until the residue was white. It was then dissolved in hydrochloric acid, evaporated to dryness, and heated for some time in an air bath, to render silica insoluble. The residue was taken up in water with the addition of a little acid, filtered, and the sulphuric acid precipitated with barium chlorid, etc., in the usual way.

(2) The method is the same as the preceding, save that 5 grams of a mixture of sodium carbonate and potassium nitrate (5:3) is used in place of the potassium nitrate. In this case the ignition must be begun very cautiously, and even with care it is difficult to prevent part of the material being thrown from the dish.

The following are the results:

TABLE V.—*Determination of SO_3 by nitric acid method.*

	Method 1.	Method 2.
	<i>Per cent.</i>	<i>Per cent.</i>
Cotton-seed meal, No. 1.....	1.22	1.12
		1.15
Cotton-seed meal, No. 2.....	1.17	1.05
	1.15	.99
Cotton-seed meal, No. 3.....	1.33	1.08
	1.28	1.08

The agreement between the duplicates was not as satisfactory as might be desired. It is seen that, for some reason, the first method gives higher results than the second, and has the further advantage that the ignition can be performed more readily.

DETERMINATION OF POTASH.

The results obtained by the different analysts for potash by the methods already described are as follows:

TABLE VI.—*Potash.*

Analyst.	Bran.		Cotton-seed meal.	
	Calcium acetate method.	Sulphuric acid method.	Calcium acetate method.	Sulphuric acid method.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
C. J. Jones, Vermont	1.21		2.03	
E. G. Runyan, Washington, D. C.	1.08	1.29	1.76	1.86
T. M. Price, Maryland	1.05	1.21	1.32	1.66
T. S. Shiver, South Carolina	1.26	1.30	1.90	1.94
R. W. Thatcher, Washington (State)....	1.22	1.22	1.73	1.98
G. S. Fraps, North Carolina	1.23	1.27	1.79	1.93
A. W. Blair, Florida	1.25		1.86	
Average	1.18	1.26	1.78	1.87

A consideration of these results leads to the conclusion that the calcium acetate method as tested—that is, when the substance is moistened with calcium acetate solution and burned in an open dish—gives results which are too low for potash. These results are not entirely unexpected. Dr. Shuttleworth, who devised this method, advises the use of a closed dish, and states that the ash prepared in an open dish contains less potash than that prepared in a closed dish.

Whether the same quantity of potash can be obtained from a plant when ignited with calcium acetate in a closed vessel as when ignited with sulphuric acid is a question that can be answered decisively only after careful experimental study. The following work by Mr. Hartwell, of the Rhode Island Station, is evidence that the calcium acetate method may not give correct results. This summary is prepared with his permission from a letter to me by Mr. Wheeler.

Analyses were made of the ash obtained from golden millet, obtained in two ways:

(1) The ash was prepared in a closed apparatus (Tucker's) with the use of 50 cc of a solution of calcium acetate, containing 0.5142 gram calcium oxid.

(2) The ash was prepared by charring the material in an open dish and extracting with water, after which the residue was incinerated.

In the first set of experiments, 0.15 per cent less potash was obtained by the use of a closed vessel and calcium acetate than by the method of charring and extracting, and similar losses of sodium and calcium appeared.

In the second set of experiments, ash was prepared by a third method also, namely:

(3) The material was charred in an open dish, extracted, and the residue incinerated with the addition of calcium acetate.

The results were as follows:

TABLE VII.—*Determination of potash as K₂O in golden millet. (Hartwell).*

	I.	II.
	<i>Per cent.</i>	<i>Per cent.</i>
(1) Calcium acetate in closed dish	1.036	1.027
(2) Charring and extracting	1.055	1.051
(3) Charring and incineration with calcium acetate	1.085	1.071

From this it would appear that there may be some advantage in the use of calcium acetate in the final incineration after the material has been extracted with water.

Perhaps by the use of a little hydrochloric acid during the extraction this would not have been the case. The calcium acetate method, even with the use of a closed dish, gives results too low. Apparently double silicates were produced, and the bases contained in these were insoluble in hydrochloric acid.

CONCLUSIONS.

In the light of this work the only possible conclusion in regard to the calcium acetate method is that it does not give correct results for potash when an open dish is used, and that even when a closed dish is used the evidence is that it may not give correct results.

RECOMMENDATIONS.

(1) It is recommended that the title of this section be changed from "Methods for the analysis of ashes" to "Methods for the determination of inorganic plant constituents."

The reason for making this recommendation is that there is no method known, at least at present, by which a plant can be burned and all of the sulphur, chlorin, and other constituents be left in the ash; that is to say, we are not able to prepare an ash which contains all the inorganic constituents of the plant.

(2) It is recommended that the method for the determination of sulphur in plants called the nitric-acid method (as modified) be adopted as a provisional method. (See (1) modification of the nitric-acid method.)

(3) It is recommended that the determination of chlorin be omitted from the methods until a method can be adopted which will approximate a correct result.

(4) It is recommended that the determination of potash by ignition of the plant substance with sulphuric acid, as described in the determination of potash in fertilizers, be adopted as an alternative method.

I have rewritten the methods of analysis relating to ashes, introducing the recommendations made above and the changes made last year, namely, "the use of calcium acetate and burning ash in some way to prevent volatilization," with the necessary minor changes. The methods are here offered as a part of this report, and without making any recommendation I submit them to the committee, to be dealt with as it deems best.

METHODS OF ANALYSIS.

VII.—METHODS FOR THE DETERMINATION OF INORGANIC PLANT CONSTITUENTS.

1. PREPARATION OF SAMPLE.

The material must be thoroughly cleaned from all foreign matter, especially from adhering soil. It is to be ground and preserved in carefully stopped bottles.

2. DETERMINATION OF CARBON-FREE ASH.

(a) *Preparation of calcium acetate.*

Dissolve 20 grams c. p. calcium carbonate in c. p. acetic acid, and dilute to a liter. Evaporate 20 cc in a platinum dish, ignite gently, then strongly, to constant weight. The dish must be weighed quickly. This gives the calcium oxid in 20 cc.

An alternative method is to dissolve marble in hydrochloric acid, evaporate, and dry to render silica insoluble, dissolve with water and a little acid, and precipitate iron and aluminum in the usual way. The calcium is then precipitated with ammonia and ammonium oxalate in hot solution, the precipitate washed well, dried, ignited, and weighed. It is then dissolved, and diluted so that 100 cc contains 1.1 grams calcium oxid.

It is best to test the purity of this reagent by making blank determinations with it.

(b) *Preparation of ash.*

Moisten 10 to 20 grams substance with 40 cc calcium acetate, dry on a water bath, and ignite, gently at first, then more vigorously. The quantity of calcium acetate used should be sufficient to prevent fusion of the ash. Some form of apparatus must

be used to prevent volatilization, either Shuttleworth's^a or Tucker's,^b or an ordinary platinum dish may be used, fitted with a cover, like that described by Wislicenus.^c The weight of the ash must be corrected for lime, carbon dioxid, and carbon.

(c) *Determination of carbon dioxid.*

Using the ash prepared in (b), liberate the carbon dioxid with hydrochloric acid in any of the usual forms of apparatus, determining the carbon dioxid evolved either by increase of weight of potash bulbs or loss of weight of the apparatus. The former method is preferred.

(d) *Determination of carbon, sand, and silica.*

The residue from the carbon dioxid determination is transferred to a beaker or evaporating dish, evaporated to dryness, and thoroughly dried and pulverized to render silica insoluble. The dry residue, etc. (See Bull. 46, Rev. Ed., Div. of Chem., U. S. Dept. Agr., p. 78, and end of section 2.)

Subtract carbon, carbon dioxid, and calcium oxid added in the form of calcium acetate from the ash, and calculate results as carbon-free ash.

3. DETERMINATION OF MANGANESE, CALCIUM, AND MAGNESIUM.

As on page 78 of the "Methods," unchanged, save that the word "ash" is inserted after "grams," line 2; add "The quantity of calcium found must be corrected for the calcium added."

4. DETERMINATION OF PHOSPHORIC ACID.

(a) An aliquot portion of the hydrochloric-acid solution, corresponding to 0.2 to 1 gram ash, is to be used for the determination by any of the methods described for total phosphoric acid in fertilizers.

(b) The determination can also be made in the plant substance as in (a), page 12 of the "Methods" for phosphoric acid, using sufficient material to give from 0.2 to 1 gram ash in the aliquot portion of the solution taken for the phosphoric acid determination.

5. DETERMINATION OF ALKALIES.

(a) An aliquot portion of the hydrochloric-acid solution (see 2) corresponding to 1.5 to 1 gram of ash is evaporated to dryness. Redissolve the residue in about 50 cc water, add milk of lime or barium-hydroxid solution, which must be perfectly free from alkalies, until no further precipitation is produced.

(b) Potash may be determined as directed for potash in organic compounds (b), page 22 of the "Methods," using sufficient plant material to get from 0.5 to 1 gram ash in the aliquot portion of the solution taken for the potash determination.

6. DETERMINATION OF SULPHUR (PROVISIONAL METHOD).

See recommendation No. 2 above, and (1) under "Modification of the nitric-acid method."

The PRESIDENT. Is there any discussion?

Mr. WHEELER. I would like to ask the referee a question, if I may. I would like to know if he has tried the method of burning in a glass tube?

Mr. FRAPS. I have not tried that. The materials that we have been working upon usually have a large quantity of organic matter, and I think it would give a little trouble.

Mr. CAMERON. Has the referee used the method of Carius?

Mr. FRAPS. The method of Carius I have never used with my plan. We constantly find a large quantity of organic matter present, and I think there would be a high pressure inside of the tube, which would

^a Exp. Station Record 11, 304.

^c Zeitschr. f. anal. Chem., 1901, 40, 491.

^b Ibid., 506.

make it very liable to explode unless you relieved the pressure by opening the tube. Further, we have none of the special tubes which must be used for this purpose.

Mr. HUSTON. How about the method of determining sulphur in coal? Have you tried the Eschka method?

Mr. FRAPS. No; I have not tried it.

Mr. HUSTON. It seems to be successful in determining sulphur in coal.

Mr. FRAPS. The trouble is the stuff is so volatile.

Mr. HUSTON. I do not think it would be any more volatile than bituminous coal.

Mr. WHEELER. We have had some difficulty. We do not know how to make the mixture of all substances in which we must determine ash. There are some materials that give no difficulty, and there are others that are extremely difficult. With chicory we could only obtain correct results for moisture by drying in a vacuum over sulphuric acid. But before we get down to the solution of this matter we must take up the question of how to determine the moisture in various kinds of material, and our methods of procedure must be adjusted to each kind of material that we are going to analyze. I am sorry to throw cold water on the matter in this way, but I feel that there is no general rule that can be applied to all organic substances.

Mr. FRAPS. I would like to state that these methods represent the best experience we have at present, and for that reason it seems advisable to use them until we can devise methods adapted to other materials and improve upon them.

The PRESIDENT. Is the referee on tannin present?

Mr. BARNARD. I was told that the referee on tannin would not be present until Saturday.

The PRESIDENT. There is one matter that might be brought up now, viz, the fixing of the time at which the committee on nominations shall make their report.

Mr. WILEY. I move that it be 3 o'clock on Friday—a special order.

The PRESIDENT. The motion has been made and seconded that the report of the committee on nominations be made the special order of business for 3 o'clock on Friday. It is adopted.

Mr. WHEELER. Are matters of general business in order?

The PRESIDENT. What is the nature of it?

Mr. WHEELER. A suggestion has been made, in view of the large number of referees and the great number of questions that are to be acted upon if possible, that the committee on recommendations of referees for next year be appointed at this meeting. I would suggest that if this general plan is adopted we begin next year on that basis. We have the committees already appointed for this meeting, and the recommendations made by the referees should be referred to the committee for report next year, and in this way every member of the

association would have a printed list of all matters that are to be acted upon placed in his hands before the end of the year, and the committee would have the whole year in which to consider the recommendations. As it is now it is very difficult for the committee on recommendations to act on the report in a satisfactory manner at the time of the meeting, while formerly it was comparatively easy. It is thought that perhaps some scheme of this kind would be desirable. Of course it would not be put into operation this year, because we have our committees already appointed, but this is brought forward now with the idea of adopting it for the future. It would not involve any change in the constitution.

The PRESIDENT. The simple action of the association would be sufficient.

Mr. ROSS. I think it is a very good suggestion to have the subjects referred to the committee, of which I happen to be a member, in advance. I know I could report to very much better advantage if I had a year in which to study them. It is a very good suggestion.

Mr. WHEELER. I move that the committees on recommendations of referees for next year be appointed at this meeting so that these committees may have time to consider the recommendations. The idea is that the recommendations will go to the committee and they will have their final recommendations printed and sent out by the committees for consideration.

The PRESIDENT. You mean that this shall take place next year?

Mr. WHEELER. It can not apply this year, but at next year's meeting. If all the recommendations go to this committee they will act upon them as soon as possible, and after being acted upon have copies printed and sent to the members of the association, so that when we come here we shall all of us have been able to consider the details of the propositions recommended by these committees, and in that way we can all act more intelligently.

The PRESIDENT. In that way there would be no reports on recommendations?

Mr. WHEELER. Not next year, under that system.

Mr. WILEY. Would it not be well in appointing a committee for next year at this session to instruct the referees to furnish the chairman and members of each committee with copies of his recommendations at least a month before the meeting of the association, so that the committee would have fully considered the recommendations before the association meets?

The PRESIDENT. The idea was to have no report next year from the committee on recommendations of referees.

Mr. WILEY. That plan might postpone some action which would be very important, whereas it seems to me that if the referees be instructed, as I have just mentioned, to furnish the committees now

to be appointed with copies of their recommendations at least a month before the meeting of the association, fully matured discussion of it could be had by the committee. At least each member could separately study these recommendations and be ready to act intelligently and without being pressed for time.

Mr. WHEELER. I am most heartily in favor of that, whether the report be made next year or a year later, but I doubt very much if it would be possible for the referees to get their reports in so that they could furnish this report to the committee. A number of referees have difficulty in getting their reports in time to write up their papers, and some have been reported after they have arrived at this meeting. I fear that can not be done.

Mr. HUSTON. It seems to me that we might make a compromise between the present method and that suggested. Sometimes there is a necessity for immediate action. Very often there is no necessity for any delay. I have been on that committee for a good many years, and part of the work can be solved very quickly, while there are some questions which require time and care to answer, but I believe that the committee should be appointed this year, if necessary, to report such things as they see fit at the next meeting. Those things which are necessary can be attended to at this meeting, but those things which are doubtful should be more carefully considered. The proposed compromise between our present method and the one suggested would be a good working solution of the problem.

The PRESIDENT. As there is no motion before us, the chair would suggest that a motion be made appointing a committee to put this affair in shape and then report later. A motion is in order to appoint such a committee if you so desire.

Mr. HUSTON. I hardly think it is necessary to appoint such a committee. If, during the present year, the committee on recommendations, any of the fifteen members you have appointed, should find some work upon which they desire more time, all they need do is to ask that they be granted more time, or that the matter be referred to the committee for the following year. This can come in with the report of the present committee and will save considerable time.

Mr. WILEY. I would like to say that if the chairmen of the committees will take these reports which the referees have handed in with them to-night, they would be able to begin at once on the work that has already been presented. The stenographer will give the reports to the committees.

Mr. HUSTON. All the reports so far go to Committee A.

Mr. Wiley at this time explained to the convention that a photographer desired to take a picture of the members the following day, and suggested that this be done at noon, which was agreed to.

The convention then adjourned, to meet the following morning at 9.30 o'clock.

MEMORIAL EXERCISES IN HONOR OF THE LATE JOHN A. MYERS.

The Washington section of the American Chemical Society invited the Association of Official Agricultural Chemists to a joint meeting in the hall of the Cosmos Club on Thursday evening, November 14. After the meeting of the chemical society was adjourned, Mr. Van Slyke took the chair and stated the object of the after meeting to be memorial exercises in honor of the late John A. Myers, one of the founders of the Association of Official Agricultural Chemists. Mr. Van Slyke spoke as follows:

We have met at this time to do honor to the memory of one of our members who had been actively identified with our association from its very beginning, who was with us one year ago in the full vigor of a splendid manhood, but who since then has been called from his earthly labors—I refer to Mr. John A. Myers. We all remember Mr. Myers for his ever genial, happy, cheerful spirit. To know him was to appreciate his sunny, hopeful disposition and to desire his friendship.

Mr. Myers was one of the charter members of our association and was president in 1889. He was seldom absent from our annual conventions and was usually active in their proceedings. His presence will be missed by those of us who knew him at all well. There is, therefore, peculiar appropriateness in holding a special session of our association to do honor to the memory of him who was so well known and generally esteemed.

Our secretary, Mr. Wiley, has prepared an address on the life and public services of Mr. Myers and will now present it.

REMARKS OF MR. H. W. WILEY.

Mr. President, members of the Washington section of the American Chemical Society, and members of the Association of Official Agricultural Chemists: It was my good fortune to have known Mr. Myers from the time we were both students until his death. I first met him in the laboratory of the Berlin University. I remember well his work there, his faithful service, and his universal popularity among the students. He displayed, even at this early date, those qualities of heart and head which continued to increase with his growth and which commended him so favorably to all those with whom he was brought in contact. I think I know something of Mr. Myers's characteristics, his strong and weak points, for the best of us can not claim exemption from imperfections.

The most prominent characteristic of his personality was his absolute sincerity. In his intercourse with his fellow-students he never had anything to conceal, nor did he ever try to present himself in a false light. After his return to the United States I kept in constant touch with his career, with his services at Butler College, Indianapolis, in the Mississippi Agricultural College, as director of the Agricultural Experiment Station of Virginia, and later in his last capacity as director of the nitrate of soda propaganda in the United States. That part of his career which we remember most vividly, perhaps, is his connection with our association. He was present

as one of its founders, and of the seventeen meetings of the association held during his lifetime he attended twelve. In this respect his example is commended to many of our directors of agricultural experiment stations who in the push of executive work sometimes seem to lose their interest in their old profession or have no time to manifest it. We congratulate our science because it has furnished so large a percentage of directors of agricultural experiment stations. Almost 50 per cent of the directors in this country and more than 70 per cent in Europe have been professional chemists. We are glad to contribute of our force to the work of direction, which we regard as of a higher character even than that of analytical practice and almost as high as that of research, but in making this contribution we hope that the interest in our own affairs will not be lost and that we shall still have the cooperation and aid of those who were formerly associated with us.

In the social affairs connected with our meetings Mr. Myers always took an important part. His good nature, his jovial appearance, and his hearty laugh were always welcome and were always missed when absent. There was no one more welcome at a dinner and no one enjoyed to a greater extent the wit and wisdom of the after-dinner speeches. After all there is nothing in life quite equal to association with a true friend. "Nil ego contulerim jucundo sanus amico," said Horace truly two thousand years ago, and the worth of this sentiment has only increased with the passing centuries.

During the last few years of his life Mr. Myers entered a new field of activity, which brought him into more intimate contact with the agricultural experiment work of this and other countries. As the representative of the syndicate controlling the output of nitrate of soda of South America, he founded in this country a propaganda unique in its scientific relations. Mr. Myers, in his capacity as the representative of this syndicate had nothing whatever to do with the sale of this useful plant food. He simply studied in a general way the needs of the great staple crops of the country for available nitrogen, and collected data and disseminated scientific articles in relation thereto. It was while engaged in this work in California and in preparing for a visit to the Hawaiian Islands that he contracted typhoid fever, which proved fatal. Although far from his home, friends were not wanting to minister to his wants, and it is a consolation to his family that nothing was left undone, either on the part of the hospital authorities or his friends, to make his days as comfortable as the disease would permit. Fortunately his wife was able to reach his bedside two days before his death, while he was still in full possession of all his mental faculties.

When it was proposed to the executive committee that some formal memorial exercises be held at the present meeting, they unanimously approved of the plan. The invitation of the American Chemical Society to join in such a meeting was forwarded to each member of the executive committee, and the following replies were received:

Permit me to state that I am thoroughly in accord with the idea that we should accept the invitation so cordially extended by the Washington section of the American Chemical Society. It is with peculiar pleasure also that I note the proposed meeting for the purpose of paying a tribute to the late John A. Myers. I sincerely feel that every member of our association will be most heartily in accord with such a plan.

Very sincerely, yours,

H. J. WHEELER.

A memorial meeting in honor of Dr. Myers will be a very proper and desirable act on the part of the Association of Official Agricultural Chemists.

Very truly, yours,

L. L. VAN SLYKE.

It meets entirely with my approval to accept the invitation of the Washington section of the American Chemical Society for our association to meet with them in the hall of the Cosmos Club on the evening of November 14, and later to hold a session of the Association of Official Agricultural Chemists in the same hall for the pur-

pose of paying a tribute to the late Dr. John A. Myers. I am especially glad that the latter meeting has been arranged, as I remember Dr. Myers most pleasantly, both as a professor in our institution and in our frequent meetings in later years. I regret my inability to be present at the meetings.

Yours, very truly,

W. R. PERKINS.

I am heartily in favor of the joint meeting of the Association of Official Agricultural Chemists with the American Chemical Society, and only regret that I shall be unable to attend this year.

Sincerely, yours,

F. W. TRAPHAGEN.

I wrote to the Permanent Nitrate Committee in London, informing them that this memorial meeting would be held, and asked for some expression in regard to the exercises. In reply to my communication I received the following letter from the secretary of the committee:

PERMANENT NITRATE COMMITTEE,
3 GRACECHURCH STREET, E. C.,
London, November 2, 1901.

DEAR SIR: My committee tender to you their cordial thanks for the opportunity you have afforded them to place on record their appreciation of the work and character of the late Dr. John A. Myers. I would say that at the meeting of my committee next after the receipt of the sad news of Dr. Myers's decease their first act was to record their sense of the great loss which they had sustained in one whose ability, energy, and devoted services during the previous three years they had so highly esteemed.

Thorough in everything he did, he died doing his duty with all his strength.

I am, dear sir, yours, faithfully,

JOSEPH HILLMAN, *Secretary.*

Unusual attention naturally attaches to the last days of a friend's life, and so the following account of the last work done by Mr. Myers will be especially grateful to his friends. This memoir was written by Mr. Charles H. Shinn, of the College of Agriculture of the University of California.

BRIEF RECOLLECTIONS OF THE LATE JOHN A. MYERS.

By CHARLES H. SHINN.

I take it for granted that Dr. Myers in his own correspondence covered the ground of these brief notes. But he was, of course, writing from his own standpoint; and as no man realizes the impression he makes upon others, the standpoint of the writer may be of some interest.

It goes without saying that the personality of Dr. Myers was to all who met him extremely pleasant. He was a man who grew upon acquaintance. He kept to a rare degree the "boyishness" which most men lose, and he was extremely sensitive to praise or blame, very high minded, and much in earnest in all he undertook. He put the interests of his business first and foremost; worked early and late to get through with his correspondence and to push business. His pleasures were simple and refined. I am constantly hearing from people who met him during our trips in California, all regretting his loss.

We left San Francisco in February, 1900, and went to the Cloverdale Citrus Fair. I had been invited to deliver an address there and incidentally to judge the fruit. I wrote the committee that if they so desired I could bring Dr. Myers with me, and they were very glad to have him come. We were met by the citizens and shown every attention. When it came to the special work of the occasion we found the hall extremely large, and very noisy. We both felt that little good would come of the effort to speak there. I asked to have Dr. Myers introduced first, and although he had said he knew "nothing about oranges," it turned out that he knew a great deal

about his subject. He gathered up sample oranges from the various exhibits and took them forward to the edge of the high platform (which was really an outjut from the balcony), and, cutting them apart, talked to the audience about "good and bad oranges," etc. He was clearly heard over most of the barn-like building, and the local newspapers spoke very nicely of his effort. I understand that a good deal was printed, and I shall try to get copies.

When it came to the judging of the fruits the local committees backed out entirely. We were forced to spend about fifteen hours on two successive days in cutting open and testing fruits according to the latest California scale. This is a modification of the American pomological scale for citrus fruits. Dr. Myers showed the greatest patience and industry, going over and over the various tests with me and checking off results, suggesting modifications, and, in short, rising to the occasion with astonishing good nature. The whole work was really thrown upon us, as the people in this district had never before had their fruit judged according to official standards.

We were together every evening and met the people of this little country district. Dr. Myers told stories, made himself entertaining to men, women, and children, and won golden opinions. He was not well, but no one would have known it from anything that he said, except that once or twice he told me that "he did not feel in good form." He was very much interested in the Sonoma Valley and its many beautiful places. He even said he would "like to bring his wife and daughter to see California again some springtime." He talked a good deal about his home and the happiness of his life. He told little stories of his boyhood. He told me about his father, his own education abroad, and a good deal that was interesting and human. We received a cordial invitation to come again next year and a special invitation was extended to Dr. Myers.

Dr. Myers then planned a trip to Oroville and the orange region in the interest of his work, and he was kind enough to say that if I would go with him he would accomplish a good deal more. We talked of it to some degree, but I found it difficult to leave my own university matters, and so I was planning to write as many letters of introduction as possible, and let him make the trip alone later.

Dr. Myers wrote me that he did not feel at all well, and so wanted to go to Paso Robles, take the hot baths, and then go south to Los Angeles, where I could join him. He seems to have had a pleasant visit at Paso Robles substation, where the foreman, Mr. Barber, drove him around the region, and he had a good rest. I then met him in Los Angeles, and we took a trip together to the Pomona substation; went around the town to some extent, and to Santa Monica. From there Dr. Myers went to Santa Ana, and saw the celery growers; then made a second trip to Pomona, and from there to Riverside and Redlands. At the latter place he arranged for some fertilizer experiments, and I believe that he had something under way at Westminster or Santa Ana.

At Pomona Dr. Myers and I dined with Mr. and Mrs. Mills and were driven around the valley. He was in better health than I had seen him for some weeks and was extremely jolly. He told stories all day, and we were trying in vain to cap each story. We called him a "Methodist minister," and told him he "should have a chicken dinner." I never saw Mr. and Mrs. Mills more amused and delighted. They have written me since that no one who ever visited them was better company and Professor Devol, of Redlands, wrote in much the same way.

While in Los Angeles we were out together and spent Sunday afternoon and evening with the family of Will E. Chapin, who is the artist and cartoonist of the Daily Times. When we returned from this visit I took the late train to San Francisco, and Dr. Myers did not go with me to the train, as had been his previous custom, because he said he "was feeling a little tired."

I had letters from him almost daily and I wrote him myself from various places.

Every one of his brief notes on business was cheerful and interesting, but I still got the impression that he was not feeling well, and on several occasions he spoke of friends of his who had passed away and of various other illnesses; then he would brace a little and say this was all nonsense and that when his cold was over he would be all right again.

I noticed with special interest from time to time the ease of his manners with the general public. He was a genuine American of the Middle West, refined by travel and study. He liked people. Naturally people liked him. It was easy for him to get along with everyone he met. Brakemen, conductors, waiters, bootblacks, and the general run of mankind liked to meet him and do things for him. He was never too tired to scatter sunshine as he went.

I had thought that I might be able to be a little more particular in some of these items, but I find that after all I have only the general impression of a thoroughly kindly and real personality mingling in the daily life of all he met. I do believe that none of this is really lost, but that the happiness he gave to his friends and to other acquaintances is a permanent part of their lives. We shall miss him and think of him often, but not without hope, nor cheerlessly. So strongly vitalized a personality, working good in its own way so many years among men, is not destroyed, although it has passed from sight. If there is one lesson among all others which we derive from religion and philosophy it is that we can go forward hoping, not fearing, and that in the hereafter we shall see face to face where now we see but blindly.

Mr. M. A. Scovell contributed a brief sketch of Mr. Myers to the Lexington (Ky.) Leader for April 12, 1901, which was in part as follows:

The friends of Dr. and Mrs. John A. Myers learn with sadness of the death of Dr. Myers in San Francisco, Cal., April 8, 1901. Only a few weeks ago he left his office in New York in the best of health for California, and now the sad news reaches us of his death, caused by typhoid fever, after an illness of only two weeks.

Dr. Myers is well remembered in Lexington, having been at one time professor of chemistry and physics at Kentucky University. While here he married Miss Minnie B. Plunkett, who survives him. * * *

Dr. Myers was an indefatigable worker in his chosen field of science, and as an investigator he stood among the first. * * * A busy man, always with some important work on hand, he was never so busy but that he could help a friend. His greeting was cordial, his hand shake came from the heart. His personality attracted and held as a magnet all who intimately knew him. Full of life, full of hope, full of ambition, he inspired, he gave cheer, and spread sunlight over all he met.

A lifelong friend and early teacher of Mr. Myers is Prof. A. E. Dolbear, of Tufts College. In answer to my inquiry concerning the early days of Mr. Myers at Bethany College, where Mr. Dolbear was then professor, he wrote me the following:

As to Myers's college life at Bethany College I remember some things very well. He came without much preparation, but was full of vigor, dead in earnest, knew what he wanted, and was willing to dig for it. At first the other fellows thought he needed discipline, and arranged to haze him. At the end of the mêlée he was not much hurt, but the other fellows needed what bandages, liniment, and court-plaster there was in the town. After that he was let alone. His rank was good in his class work, and he never got tired. In the chemical laboratory he was painstaking, and learned pretty thoroughly what was the meaning of the work he did. At the table he was hearty, and to the dismay of the boarding house would call for more butter and eggs when the plates had been emptied. Such things showed he could be trusted to look after his health.

I do not remember that he took part in any of the college sports, nor do I think he had any of the mild vices. After his graduation at Bethany he took a course of experimental physics at Tufts College, and then went to Europe to study chemistry. He was altogether honorable, and I wish he might have lived to patriarchal age.

I have seen very little of him for the past twenty years, and doubtless you know far better of his work. I should be glad to add to this, but the time I knew him was nearly thirty years ago.

An interesting letter from Gen. Stephen D. Lee, president of the Mississippi Historical Society, and former president of the Agricultural and Mechanical College of Mississippi, gives a short account of Mr. Myers's service in that institution:

Prof. John A. Myers was elected to the chair of chemistry in the Agricultural and Mechanical College of Mississippi in 1882, and filled the place for eight years. He was a most accomplished chemist, superintended the erection of the chemical building, and instructed all classes to the great satisfaction of the board of trustees and the president. During the time he was at the college several young men graduated in his department with honor, and are now filling prominent positions in several States. Among them may be mentioned Prof. H. H. Harrington, of the Texas Agricultural College, and B. W. Kilgore, director of the North Carolina Experiment Station. Professor Myers was an accomplished gentleman and a most industrious and painstaking worker.

Mr. President, there are other gentlemen here who desire to make some tribute to the memory of Mr. Myers, and I reserve the rest of my remarks until they have been heard.

REMARKS OF MR. B. B. ROSS.

The first intimation I received of the death of Mr. Myers came to me through Mr. Wiley, whom I met in London during the past summer, and it is needless to say that I was greatly shocked at the sad intelligence, as I had anticipated calling on him at his New York office upon my return to this country.

Only a few months before going abroad I had the pleasure of entertaining Mr. Myers at my own home, and at that time I well remember he exhibited in a marked degree those sunny, genial, and amiable traits which were so strikingly characteristic of the man, and to which such happy references have been made in the letters of friends to which you have just listened.

My acquaintance with him did not extend over so long a period as that of some who have already spoken, and dates from the third annual convention of the Association of Official Agricultural Chemists, the first which I had had the opportunity of attending. I remember quite well that, although an almost total stranger in that gathering, I received a most kindly greeting and welcome from Mr. Myers, and when, with some trepidation, I had ventured to take part in the discussion of the association methods, I now recall with pleasure the fact that upon that occasion, as well as at subsequent conventions, his kindly words of encouragement to myself and other young men in attendance did much to make us feel at ease and at home in the meetings of this body.

From that date up to last year I met him at frequent intervals, and as my opportunities for knowing him increased I became more and more impressed with those traits and characteristics which endeared him to his many friends.

This is the first meeting for many years that I have missed his well-known presence in this gathering, and, although he is gone from us, the recollection of his genial nature, his kindly greeting, and his friendly hand clasp will linger with me as a pleasant memory.

REMARKS OF MR. KILGORE.

It was my good fortune to know Mr. Myers well. For eighteen years he was to me one of the best of friends, and I acknowledge publicly now, as I have done frequently in a private way during his lifetime, my indebtedness to him for many favors and kindnesses. As a student in college I came under the influence of his instruction as professor of chemistry in the Mississippi Agricultural College. In the capacities of student, assistant, and guest in his home the different phases of the life of the man in whose memory we have met to-night became familiar matters to me, and I therefore feel specially competent to speak concerning him who has so recently gone

from us, leaving great sorrow in the hearts of his many friends, but the most pleasant memories of a useful life, full of service to his fellow-man and to his chosen line of work. I have always regarded Mr. Myers as one of the best of teachers, because he possessed the power and capacity in a marked degree of inspiring and obtaining work from students where others could not get it. It was either work or get out of his classes, and in consequence of this not a few of his students did not regard him pleasantly during their early student days, but by the time they finished their college work and were ready to enter seriously into the realities of life the early feeling of dislike had given place to one of high regard. They now knew the man as he really was, and that, though seemingly hard, he had all along been doing the very best thing for them. He was eminently a success as a teacher, and I have had occasion recently and since his death to speak with some of his former students, who have spoken of him in accord with the remarks I have made.

He did a great service in assisting in the organization and development of the Agricultural College of Mississippi, under the presidency and leadership of that magnificent type of Southern man, Gen. Stephen D. Lee. Mr. Myers was an organizer and leader of undisputed ability, and it was always known where he stood in matters of discipline, policy, and right. Of this work General Lee has spoken, in his communication to the secretary of this association, in unmistakable terms, and of course with authority. This phase of our much-beloved friend's life—that of teacher and college man—I think few, if any of you, are familiar with, and I thought that it would be of interest for me to refer to it here, though I have done so in but a brief and very imperfect way, and I need not say to you, gentlemen, that it brings to me great sorrow.

Mr. Wiley has spoken of Mr. Myers's early connection and earnest and continuous interest in the organization, progress, and growth of the Association of Official Agricultural Chemists to the time of his death. A great deal of this is a personal memory to most of you. I feel specially kindly to him for the help he gave me as a young worker in this association, and for the encouragement received from him and from other older workers, to take the modest part I have in the association work. They have encouraged us to speak out in meeting on all subjects on which we felt we had experience and information that would throw light on the varied and important work that was being done. This spirit of making all feel that they were at home and a necessary part of the work that was being done has helped to make this association what it is, and we love it, and revere and honor the memories of the men who have been its active workers. Mr. Myers's important and valuable work in connection with the experiment-station movement in this country, in which we are all so much interested, has been spoken of, and besides is a matter of memory. For these reasons it is not necessary for me to review this phase of his life work.

We see, though, from the various lines of work to which he has turned his hand with unflinching success, that his life was an exceedingly active one, and he departed it in the midst of great usefulness and in the prime of strong and vigorous manhood, to be mourned by a host of admiring and loyal friends, and most tenderly by a devoted wife and loving daughter. May God bless the broken home and sanctify to the good of all who knew him the memory of his life work.

This, Mr. President, has been a very sad duty for me, and it has been but imperfectly done. To give more appropriate and official expression to our regard and estimate of the services of our departed friend and coworker, I move that a committee be appointed to draft suitable resolutions to be incorporated in the proceedings of this meeting.

This motion being carried, Messrs. Wiley, Kilgore, and Voorhees were named as the committee to draft said resolutions.

REMARKS OF MR. VOORHEES.

I can only echo the sentiments already expressed by my confrères, Messrs. Wiley, Kilgore, and Ross, though I can not let the opportunity go by without giving voice to my deep admiration and love for our departed associate. His was a life that was helpful to all with whom he came in contact. My acquaintance with him began about 1882, when, after his return from Germany, he visited the experiment stations of the East, and the respect formed for him then grew, as my acquaintance ripened, into friendship and love. He was helpful always, and I well remember his words of encouragement and assistance when I first attended the meetings of this association. He it was who first met me and insisted that I should give to the meeting the results of the study of methods which he knew had been carried out at our station, and always at any of our meetings he took pains to bring forward and encourage young men. In later years it was my privilege to come into frequent contact with him, both in a social and a business way, and the more I knew of him the more I admired him for his generous, helpful character. He looked on the bright side of life, was always cheerful and hopeful, and yet forceful. He made things go by sheer force of character. As an association we have lost an able and faithful coworker, and as individuals we have lost a true and tried friend.

Mr. WILEY. Mr. President, I wish to present the following biographical sketch of Mr. Myers:

BIOGRAPHICAL SKETCH OF JOHN A. MYERS.

John Alva Myers, A. M., Ph. D., was born May 29, 1853, on a farm near West Liberty, Ohio County, W. Va., where he resided until after he completed his college course, working upon the farm during his college vacations, and knowing nothing of city life until after his graduation. He prepared for college at the West Virginia State Normal School at West Liberty, and graduated at Bethany College in the A. B. course in June, 1875. During his last year at college he was placed in charge of the chemical laboratory of that institution, which at that time was one of the most complete in the Southern States. After graduating he remained one year in that institution, taking post-graduate studies and teaching analytical chemistry. He was elected professor of chemistry and physics in Butler University, near Indianapolis, Ind., where he organized and fitted up their chemical and physical laboratories. At the end of one year he resigned his position to continue his studies in the universities of Germany, where he remained three and a half years, studying and carrying on original investigations in the chemical laboratories at Göttingen, Breslau, and Berlin, under the immediate guidance of some of the most celebrated professors of Europe. His vacations were used for traveling, which enabled him to travel over nearly all of Europe, through Egypt, Palestine, and portions of Turkey and Greece. During his last year in Germany he was elected professor of chemistry and natural history in the Kentucky University, which he reorganized, and where he added materially to the equipment and efficiency of the chemical laboratory.

From Kentucky he was invited to the Agricultural and Mechanical College of Mississippi. Here he planned, built, organized, and equipped their splendid chemical laboratory, one of the best in the South, and also organized the work of the State chemist, being the first State chemist of Mississippi. He also organized the chemical work of their agricultural experiment station, of which he was the first chemist, and in conjunction with Prof. F. A. Gully, the professor of agriculture in that institution, he organized the first Farmers' Institute ever held in the State of Mississippi. During his stay of seven years in that State he saw the agricultural interests of the State materially advance through these channels.

In 1888 he was invited by the board of regents of the West Virginia University to return to his native State and organize the West Virginia Agricultural Experiment Station. The entire organization of the experiment station, the planning of its buildings, their equipment, the organization of the work, and the assembling of the magnificent collection of apparatus, libraries, etc., were also effected under his direction. He also organized the West Virginia Agricultural College, of which he was dean, and the farmers' institute work in that State, as well as the work of the State chemist of West Virginia. After continuing in this work for ten years, Mr. Myers was appointed manager of the Propaganda for Nitrate of Soda in the United States, since which appointment there have been added to the territory under his charge Cuba, Porto Rico, the Sandwich Islands, and the British Provinces of North America.

Mr. Myers was one of the founders and ex-presidents of the American Association of Agricultural Chemists, which is everywhere recognized for the accuracy of its scientific methods. He was one of the founders of the Association of Agricultural Colleges and Experiment Stations. He served as a member of its executive committee for a number of years, and was also one of its vice-presidents. He has also served on various State boards and was frequently commissioned by the governor of West Virginia as a delegate to National Agricultural Development or Immigration associations.

Mr. Myers was also a member of the Berlin Chemical Society, the American Chemical Society, and Fellow of the American Association for the Advancement of Science.

Mr. Myers's scientific work has been published largely in the form of reports, bulletins, pamphlets and articles in the agricultural press, and was received with such favor that portions of it have been translated for use in Germany and Japan. His work has been preeminently one of organization, in which he was so thorough, energetic and effective that the laboratories left by him have, in several cases, scarcely been changed since he left them.

MR. WILEY (continuing). Mr. Myers died at Waldeck Hospital, San Francisco, on April 8, 1901, of typhoid fever contracted either on his way to or during his stay in California. His body is buried in the cemetery in Lexington, Ky., and a movement is on foot among his friends to erect a proper monument to his memory.

I can not forbear, Mr. President, on this occasion to call attention to a loss which agricultural science has lately suffered in the death of one of our colleagues in Europe—Prof. Max Maercker, of the University of Halle. Maercker's personality was to me so very much like that of Myers—whole souled, hearty, the best of companions, the most hospitable of hosts, and the most agreeable of guests. Many of us remember his long visit to Chicago during the time of the World's Fair, and I recall with special pleasure my association with him during his visit to Washington and my stay with him at Halle in 1898. I remember him so well when, late in the evening, perhaps almost morning, he stood upon the steps of his residence and called his last farewells to me and his best greetings to his brethren in the United States. His death at the early age of 59 occurred on the 18th of October at Halle. I wished to associate his name with that of Mr. Myers.

The operator of the lantern will now throw upon the screen an excellent portrait of Mr. Myers, taken from one of his latest photo-

graphs, which will be reproduced as the frontispiece in the next volume of the proceedings of this association. I will ask that all persons present rise and stand in silence for a few moments in this last official tribute to his memory.

RESOLUTIONS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS IN COMMEMORATION OF THE LATE JOHN A. MYERS.

Whereas the members of this association have learned with profound regret of the death, on April 8, 1901, of John A. Myers, one of its founders, whose services to the association have been faithful and efficient, and whose genial personality was beloved by all his colleagues, be it

Resolved, That we place on record in the files of our proceedings an expression of our appreciation of the aid which his unwavering interest and cooperation have been to us in the past, and the inspiration which his memory will be to us in the future;

Resolved, further, That a copy of these resolutions be transmitted to Mrs. John A. Myers with an expression of our personal association in the grief which she has experienced in his premature death.

H. W. WILEY,
B. W. KILGORE,
E. B. VOORHEES,
Committee.

SECOND DAY.

FRIDAY—MORNING SESSION.

President Van Slyke called the meeting to order at 10 a. m.

Mr. Kilgore reported for the committee appointed to wait upon the Secretary and Assistant Secretary of Agriculture as follows:

The committee has performed its duty and found the Secretary out of town, but his secretary promised to give the message to him, and it is likely that he will visit the association. The committee also called upon the Assistant Secretary of Agriculture and extended to him an invitation to attend the proceedings of the convention.

On motion of Dr. Bigelow, the report was approved and the committee discharged.

The PRESIDENT. We will take up now the report on liquor and food adulteration.

REPORT ON LIQUOR AND FOOD ADULTERATION.

By W. D. BIGELOW, *Referee*.

I shall bear in mind the suggestion made by the president in his opening address. It was, in brief, I believe, that the least important portions of the report be omitted in reading. So I shall not read this morning the whole report which the committee has to present. We shall probably save several hours by it. You are probably all aware that in the address of the retiring president, Mr. Kilgore, a year ago, it was suggested that the work of the referee on liquor and food adulteration be increased, or rather that the subject be divided into a number of classes, each of which should be put in charge of one man to prepare methods for the examination of foods. This was acted on, with the result that fifteen classes of foods were arranged and associate referees appointed for each one, or, rather, each one put under the direction of one member of the association. In some cases one man was given more than one class. Unfortunately it happened, after the meeting had adjourned and the men were notified and asked to cooperate with us, that several were unable to do so, and it was necessary to make changes. This was very unfortunate because of the fact that some on whom we had counted for excellent work, who were fully qualified and very familiar with the subjects they were asked to take care of, found they did not have time to undertake it at all. The work was taken up, however, others were appointed in their places, and in thirteen instances reports have been prepared.

Now, it must be borne in mind that these reports are only provisional, and in many cases are imperfect and incomplete. It has been our idea that each author should include only the methods with which he was perfectly familiar, and which he could personally recommend—not always satisfactory, but in all cases the most satisfactory to his knowledge—and usually inherent errors in the methods are stated and the degree of excellence is given. In several cases a great deal of time has been devoted to the study of methods by the various referees, often covering several months, so

that several of the papers presented represent a fairly thorough study of the subject. In other cases, owing to lack of time, the referees have attempted to cover but part of their subjects, and it is intended, of course, that other portions shall be added later. In several instances—four, I believe—no report has yet been presented, because it was impossible for those in charge of the work to consider it sufficiently to offer reports. One report which has not been received yet will be prepared—that on infants' and invalids' foods—and published with the others. In all, 106 printed pages have been prepared. As each set of methods was completed it was distributed to a mailing list of 106, including each experiment station and agricultural college in the country, and, as far as we were acquainted with them, to every board of health and dairy and food-commission laboratory. Ordinarily it has been our custom to send but one copy to each laboratory, with the request that the recipient bring it to the attention of his associates. The result has not been all that we hoped. The criticisms have not been as extensive as we desired, and yet a number of minor changes have resulted from them, and these have been very helpful.

The committee met day before yesterday and yesterday to consider the full subject in as great detail as possible. I think there were only two or three methods—and those minor and unimportant—that were stricken out. In one instance this was due to the fact that since the methods were offered in the first place new work has been done and published by others and the methods there suggested taken up by the referee in charge, confirmed, found more satisfactory than his own, and substituted. In one other case it was thought by practically all of those who met and considered the matter that one of the methods given could be substituted by a somewhat similar one to advantage, and, since there was no objection to that, it was done. I think there was no method of those suggested cut out altogether.

We have put together here temporarily the methods that were distributed for criticism. The work was done as quickly as possible, with very little revision, the idea being merely to get them in shape for criticism. The proof was not read carefully, and there was a large number of typographical errors. Of course, all that will be corrected in the permanent report. It is not the idea that these shall be preserved, but merely criticised, and that the amended work shall be published in the near future.

I would call attention especially to one feature, which we regard as important, in which these methods differ from the regular methods of the association, and that is that in nearly all cases we have given the original reference for each method, with the name of the originator of the method. In some cases it was difficult to find the author of the method, but this was done as far as possible, and the committee thinks it would be an excellent plan to follow throughout the publications of the association.

One word regarding the practice of distributing these reports. It seems to me that it might be adopted to advantage by all of the referees of the association. All referees' reports could probably be completed a month earlier than they are. As a matter of fact very often they are not handed to the secretary until a month or two after the time of meeting. Now, if they were printed and distributed before the meeting of the association they could be discussed intelligently.

With regard to the correspondence that has come in I can say that we have been very much gratified by getting in touch with a great many chemists who were interested in food work whom we did not know at all, and we seem to have been brought into closer cooperation with them, and I believe it has added to this branch of the work of the association.

In closing I wish to express my gratitude to all of my associates for the hearty and cordial cooperation which I have received throughout, and while it is very difficult to particularize or specify, perhaps it is only due to the local associates, Messrs. Munson and Tolman, to recognize also their cooperation in the work in getting the reports ready for the printer, in addition to their own contributions, which were important;

We have two criticisms or recommendations to make. In the first place, that the referee be known as the "referee on food adulteration," instead of on "liquor and food adulteration," as he is now called. You will recognize the fact that the liquor work is now a comparatively unimportant portion of the work of the referee, and there is no reason for specifying it in his title. Second, that the methods outlined by the referee and associates—as amended in our conference yesterday—be published separately as provisional methods of the association, under the authors' names.

The suggestions received in correspondence, when they are not adopted by the referees and the methods changed accordingly, will be printed in the publication referred to above as an appendix. Very often these suggestions are of considerable importance; new methods may be suggested which are considered of value, and yet if the referee has not tried them himself they can not well be included in his report. In some cases entirely new methods have been outlined. These may be of value, but we do not feel that we should assume the responsibility of them.

The PRESIDENT. Are there any special papers to be presented on the subject? We can easily imagine that this outline contains about everything that can be given at present. Discussion is in order on the subject. Is there anything more? If not, we will have the report on nitrogen.

Mr. Ferris then read the report on nitrogen, by Mr. Perkins.

REPORT ON NITROGEN.

By W. R. PERKINS, *Referee*.

The work of the referee during the past year has embodied only a repetition of the trials of the neutral permanganate (Street) and alkaline permanganate (Jones) methods for determining the availability of organic nitrogen.

Samples of fish, tankage, garbage fertilizer, dried blood, cotton-seed meal, and a mixed fertilizer the nitrogenous portion of which was furnished from cotton-seed meal, blood, and garbage constituted the samples distributed for the work.

In compliance with the request from different laboratories sixteen sets of these samples were sent out. Reports were received from eight laboratories embracing the work of twelve analysts.

The following letter, describing the samples and giving directions for the work, was sent out with the sample:

AGRICULTURAL COLLEGE, MISS., June 27, 1901.

DIRECTIONS FOR WORK ON NITROGEN FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1901.

Six samples have been prepared for the work on nitrogen, as follows: No. 1, fish; No. 2, tankage; No. 3, garbage fertilizer; No. 4, dried blood; No. 5, cotton-seed meal; No. 6 is a mixture containing 100 parts of dried blood, 200 parts of cotton-seed meal, 600 parts of garbage, and 900 parts of acid phosphate.

TOTAL NITROGEN.

Determine total nitrogen by Kjeldahl method.

AVAILABLE NITROGEN.

Determine by the following methods:

(1) *Neutral permanganate method (Street)*.—Into a 400-cc Griffin beaker, low form, weigh an amount of sample containing approximately 0.075 gram of nitrogen. Digest this with 125 cc of permanganate of potash solution (16 grams of potassium permanganate to 1,000 cc of water) in a steam or hot-water bath for thirty minutes. Have

the beaker let down well into the steam or boiling water and keep closed with a cover glass, stirring once or twice with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water, and filter through a heavy 15-cm folded filter. Wash with cold water till total filtrate amounts to 500 cc. Dry and determine nitrogen in residue by Kjeldahl method. Wash sample No. 6 before digestion, as in determining water soluble phosphoric acid.

(2) *Alkaline permanganate method (Jones).*—Weigh out an amount of sample containing approximately 0.045 gram of nitrogen, and transfer to a 600-cc distillation flask. After connecting with condenser to which the receiver containing standard acid has been attached, digest with 100 cc of alkaline permanganate solution (16 grams of potassium permanganate and 150 grams of sodium hydrate dissolved in 1,000 cc of water) for 30 minutes below the boiling point. Then boil slowly until the distillation is completed.

W. R. PERKINS, *Referee.*

FRED W. MORSE, *Associate Referee.*

The results of the work by the different analysts appear in the tables below:

TABLE I.—*Total nitrogen.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. B. Robb, College Park, Md.	8.42	7.53	2.93	13.62	6.39	2.40
C. H. Jones, Burlington, Vt.	8.34	7.40	2.91	13.56	6.30	2.38
C. D. Holley, Orono, Me.	8.35	7.51	2.96	13.71	6.40	2.48
O. W. Knight, Orono, Me.	8.38	7.48	3.02	13.69	6.42	2.46
T. M. Price, College Park, Md.	8.58	7.49	3.06	13.74		2.45
J. W. Ames, Wooster, Ohio.	8.30	7.56	2.98	13.81	6.41	2.57
W. R. Perkins, Agricultural College, Miss.	8.34	7.41	3.01	13.59	6.35	2.46
E. B. Ferris, Agricultural College, Miss. ..	8.30	7.47	3.02	13.56	6.37	2.44
W. H. Scherffius, Lexington, Ky.	^a 8.23	^a 7.38	^a 2.93	^a 13.56	^a 6.36	^a 2.40
Do.	^b 8.35	^b 7.47	^b 3.04	^b 13.75	^b 6.45	^b 2.45
J. P. Street, New Brunswick, N. J.	8.22	7.48	2.99	13.44	6.31	2.54
F. B. Carpenter, Richmond, Va.	8.34	7.50	3.06	13.70	6.37	2.42
S. H. Shieb, Richmond, Va.	8.36	7.49	3.08	13.69	6.43	2.35
Average.	8.35	7.47	3.00	13.65	6.37	2.44

^a Digested three hours.

^b Digested seven hours.

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. B. Robb, College Park, Md.	0.66	0.38	1.43	0.29	0.34	0.60
	.70	.41	1.42	.25	.31	.70
	.60	.38	1.46	.39	.24	.70
C. H. Jones, Burlington, Vt.	.85	.83	1.49	1.78	.94	1.05
C. D. Holley, Orono, Me.	.89	.54	1.62	.96	1.37	1.30
	.89	.56	1.62	.96	1.37	1.30
	.89			1.00		
O. W. Knight, Orono, Me.	1.09	.69	1.71	1.06	1.39	1.46
	1.23	.70	1.71	1.02	1.43	1.38
	1.23	.70		.95	.37	1.35
				.95		1.40
T. M. Price, College Park, Md.	.71	.54	1.54	.78		.81
J. W. Ames, Wooster, Ohio.	.19	.46	1.61	.25	.33	1.01
W. R. Perkins, Agricultural College, Miss.	.80	.63	1.64	.96	.79	.71
	.64	.41	1.68	.81	.82	.73
	.67	.74	1.52	.64	.85	.70
	.84		1.54	.74	.86	.65

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash—Continued.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
W. R. Perkins, Agricultural College, Miss.	-----	-----	1.61	-----	0.65	0.41
	-----	-----	1.62	-----	-----	.40
	-----	-----	1.58	-----	-----	.56
	-----	-----	1.54	-----	-----	.51
E. B. Ferris, Agricultural College, Miss.	0.67	0.54	1.56	0.59	.52	.59
	.59	.57	1.50	.47	.71	.56
	-----	.53	-----	-----	-----	.47
W. H. Scherffius, Lexington, Ky	.60	.32	1.51	.36	.46	.80
	.91	.35	-----	.48	.41	.60
J. P. Street, New Brunswick, N. J	.82	.55	1.69	1.03	1.07	.65
	.84	.60	1.72	1.07	1.12	.67
S. H. Shieb, Richmond, Va.	.64	.52	1.83	.48	.79	.84
	.67	.52	1.87	.66	.77	.82

TABLE III.—*Availability of organic nitrogen by the neutral permanganate method.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. B. Robb, College Park, Md.	92.06	94.95	51.19	97.87	94.67	73.33
	91.68	94.55	50.85	98.16	95.30	70.83
	92.87	94.95	50.17	97.13	96.24	-----
O. H. Jones, Burlington, Vt	89.75	88.76	48.59	86.86	85.06	55.97
C. D. Holley, Orono, Me	89.34	92.81	45.27	93.00	78.60	^a 47.58
	89.34	92.53	45.27	93.00	78.60	^a 47.58
	89.34	-----	-----	92.71	-----	^a 47.58
O. W. Knight, Orono, Me.	86.99	90.64	43.37	92.25	78.34	^a 40.65
	85.32	90.77	43.37	92.55	77.72	^a 43.90
	85.32	90.77	-----	93.06	78.66	^a 45.12
	-----	-----	-----	93.06	-----	^a 43.09
T. M. Price, College Park, Md.	91.72	92.79	49.67	94.32	-----	66.93
W. R. Perkins, Agricultural College, Miss.	90.40	91.50	45.51	92.93	87.55	^b 71.13
	92.32	94.46	44.18	94.03	87.08	^b 70.32
	91.86	90.01	49.50	95.29	86.61	^b 71.54
	89.92	-----	48.83	94.55	86.45	75.57
	-----	-----	46.51	-----	89.76	^c 83.33
	-----	-----	46.17	-----	-----	^c 83.73
	-----	-----	47.50	-----	-----	^c 85.36
	-----	-----	48.83	-----	-----	^c 79.26
J. W. Ames, Wooster, Ohio	97.68	93.96	45.98	98.19	94.86	60.78
E. B. Ferris, Agricultural College, Miss.	91.92	92.77	48.34	95.64	91.83	75.81
	92.89	92.36	50.33	89.16	88.85	77.04
	-----	92.90	-----	-----	-----	-----
W. H. Scherffius, Lexington, Ky	92.81	95.71	50.32	97.38	92.86	67.34
	89.10	95.31	-----	96.50	93.64	75.51
J. P. Street, New Brunswick, N. J.	90.00	92.60	43.20	92.30	83.20	74.40
	89.70	92.00	42.10	92.00	82.20	73.60
S. H. Sheib, Richmond, Va.	92.34	93.05	40.58	96.49	87.71	64.25
	91.86	93.05	39.28	95.18	88.02	65.10
Average	90.69	92.74	46.50	94.14	91.09	69.85

^aOmitted from the average.^bFilter paper digested with sample.^cSample washed off of paper with permanganate solution; omitted from the average.

TABLE IV.—*Results on digestion of organic nitrogen with alkaline permanganate of potash.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. B. Robb	5.72	5.58		6.42	3.64	1.42
	5.70	5.64		6.34	3.82	1.39
	5.78	5.60			3.80	1.43
C. H. Jones, Burlington, Vt	5.10	4.30	0.51	8.99	2.72	.71
C. D. Holley, Orono, Me.....	5.59	4.70	.66	8.56	2.58	.77
	5.59	4.70	.65	8.56	2.58	.72
	5.59	4.73				.72
O. W. Knight, Orono, Me.....	5.51	4.68	.60	8.33	2.57	.77
	5.43	4.58	.59	8.39	2.57	.75
	5.47	4.58		8.39		.71
T. M. Price, College Park, Md	5.54	4.14	.68	6.49		.72
W. R. Perkins, Agricultural College, Miss	5.65	4.92	.67	9.30	2.98	.71
	5.67	4.90	.73	9.54	3.16	.86
	5.63	4.87	.72	9.17	2.98	.78
	5.50		.69	9.35	2.96	.81
	5.78			9.04	2.64	.86
						.88
E. B. Ferris, Agricultural College, Miss.	5.77	5.25	.74	9.04	3.22	.79
W. H. Scherffius, Lexington, Ky	2.91	3.19	.77	7.28	4.34	.60
	4.76	4.04	.47	8.40	2.91	.60
	3.29	2.94		6.87	2.21	.43

TABLE V.—*Availability of organic nitrogen by the alkaline permanganate method.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
J. B. Robb, College Park, Md.....	68.01	74.10		47.13	56.96	* 59.16
	67.77	74.90		46.54	59.46	* 49.58
	68.40	74.36			57.78	* 57.91
C. H. Jones, Burlington, Vt	61.15	58.17	17.73	66.30	43.17	29.79
C. D. Holley, Orono, Me	66.94	62.58	22.29	62.44	40.31	31.05
	66.94	62.58	21.96	62.44	40.31	29.03
	66.94	62.97				29.03
O. W. Knight, Orono, Me.....	65.75	62.57	19.86	60.85	40.03	31.30
	64.80	62.58	19.54	61.29	40.03	30.49
	62.27	61.23		61.29		28.86
T. M. Price, College Park, Md	64.56	55.27	22.22	* 47.23		29.38
W. R. Perkins, Agricultural College, Miss	67.74	66.39	21.30	68.43	47.20	28.40
	67.98	66.12	24.30	* 72.47	50.00	30.50
	67.50	65.72	22.92	67.47	47.20	32.92
	65.95		23.92	68.80	46.61	34.95
	69.30			66.52	41.57	35.77
						32.52
E. B. Ferris, Agricultural College, Miss.	69.51	70.28	24.50	66.66	50.54	32.37
W. H. Scherffius, Lexington, Ky	* 34.80	* 42.59	* 25.00	* 53.10	* 67.49	* 24.49
	* 56.93	* 53.93	* 15.26	61.34	45.25	* 24.49
	* 39.35	* 39.25		* 50.16	* 34.37	* 17.55
Average	66.20	63.74	21.86	64.30	44.35	31.09

* Omitted from the average.

TABLE VI.—*Computed and determined availability of nitrogen in sample No. 6.*

	Total N.	Neutral permanganate method.		Alkaline permanganate method.	
		Undissolved N.	Availability of N.	N. removed.	Availability of N.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Computed	2.46	0.82	66.66	1.02	41.46
Determined	2.44	.71	69.85	.76	31.09

COMMENTS BY ANALYSTS.

J. M. Bartlett.—From the study of results I think that the neutral permanganate method promises much better than the alkaline permanganate; although, perhaps, comparative results with the alkaline show as great differences as the neutral, still the percentages of available nitrogen are not nearly up to what soil tests show.

J. B. Robb.—In the alkaline permanganate method I added some pumice stone before distilling to prevent the contents of the flask from bumping.

John Phillips Street.—I followed your directions in every particular, except that I was contented to stop with 400 cc of washings. Past experiments which I have made show that there is a very small amount of nitrogen in the total filtrate, by far the greater part being driven off by the permanganate during the digestion. The prolonged washing suggested, therefore, seems to be unnecessary. Four or five thorough washings should suffice.

F. B. Carpenter.—Mr. Sheib reports that he was unable to get any satisfactory results by the alkaline permanganate method. He experienced great difficulty in distilling the nitrogen after digestion on account of violent bumping, and in some instances excessive foaming. The neutral permanganate method seemed to give very much more satisfactory results in case of the class of materials which were included in your samples. I do not believe, however, it is of very much value as a standard for comparison with the actual availability of the compounds in the soil. I have seen some results by this method on organic ammoniates which had been acidulated with sulphuric acid which were lower than the material showed before the treatment.

A. M. Peter.—In regard to the total nitrogen you will notice that there is a perceptible gain by digesting a longer time. It would seem that six hours is the time needed for these samples. While the average fertilizer may give up its nitrogen in about three hours' digestion, I do not think that substances high in nitrogen will always do so, and I think our official directions ought to be so modified as to call attention to this point, which is quite likely to be overlooked. It will not always do to stop the digestion when the solution has become colorless, or nearly so, or a light straw color. I believe this point has been worked on over in Germany, and the evidence obtained is somewhat conflicting, though much of it tended to show that long digestion was necessary where extreme accuracy is required, and that the use of P_2O_5 in the sulphuric acid hastens the results. An experiment I made some time ago upon tobacco stems may be of some interest in this connection, though involving the method as modified to include nitrates. I used 1.4 grams of stems, 0.5 gram of salicylic acid, 30 cubic centimeters sulphuric acid, 2.5 grams of sodium thiosulphate, and 0.5 gram metallic mercury.

The following percentages of nitrogen were obtained:

	Per cent.
1. Digestion as usually done here, not timed, average of 2.....	2.60
2. Digestion stopped as soon as colorless	2.43
3. Digestion continued much longer, not timed.....	2.71
4. Digestion stopped at end of 2 hours, mean of 2	2.74
5. Digestion stopped at end of 3½ hours, mean of 2	2.80
6. Digestion stopped at end of 5 hours, mean of 2	2.82

In this instance it seems the digestion had to be continued considerably after becoming colorless, and required about three and one-half hours.

DISCUSSION OF RESULTS.

No comments on the results in Table I are necessary, further than calling attention to the differences existing between the highest and the lowest determinations on samples 1, 4, and 6. The results sent by Dr. Peter, together with his comments, indicate a probable cause of such variation. My own observations on such material as blood, fish, and cotton-seed meal have led me to the same conclusion, and I suggest the advisability of placing such a precaution in the method, or else that the matter should receive the attention of the association, through its referee, on this subject. By reference to Table II we observe that with samples Nos. 1, 2, and 3 the figures are comparatively uniform, with the exception of four results. In samples with as high a percentage of nitrogen as Nos. 1 and 2 contain, such variations do not count for so much on the per cent of nitrogen available, and on the whole, I think, are quite satisfactory. With samples Nos. 4, 5, and 6 the differences are more marked, but also assume very satisfactory proportions after rejecting a few of the higher ones. With regard to this method we will note two especial conditions in the manipulation, one or both of which may account for the varying results in this table.

1. After digesting and filtering the sample should be washed to a uniform extent.

2. All fertilizers containing acidulated material should be washed till free of acid before digestion. It is undoubtedly true that much of the nitrogen by this method remains in the filtrate and is not converted into ammonia by the permanganate. This being the case, it is evident that insufficient washing will leave some of the filtrate in the paper or sample and consequently increase the amount of undissolved nitrogen. As evidence on this point, mention was made in the report of last season of the fact that the soluble phosphoric acid of acid phosphate would precipitate nitrogenous compounds from the filtrates. Further work has shown that acids, both organic and mineral, will act in the same manner, throwing down a copious precipitate of these protein compounds. As bearing further on this point, a full set of the official samples were digested as usual, and instead of adding 100 cc of water before filtering, 100 cc of 1 per cent hydrochloric acid was added, and the filtration and washing continued as usual. The nitrogen in the results was as follows:

TABLE VII.—*Digestion of organic nitrogen with neutral permanganate of potash, and additions of hydrochloric acid.*

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Nitrogen in residue	3.00	2.80	1.90	4.15	4.26	1.67
Nitrogen removed.....	5.34	4.51	1.11	9.44	2.09	.79
Total nitrogen removed	63.95	60.37	37.00	69.15	32.18	32.37

The results in the first trial are very much higher than those obtained in the regular way. In fact, the portions of nitrogen removed by the process bear a very striking resemblance to the so-called available nitrogen by the alkaline permanganate method as shown in Tables IV and V. Mention is made of this in order to emphasize the importance of uniformity in washing the residue, and also to call attention to the results obtained on acidulated goods if the free acid is not removed before the digestion of the sample with permanganate. The results by the alkaline permanganate method are very unsatisfactory when considered as a whole, though an examination of the results of the different analysts, with one exception, indicates that entirely satisfactory results might be obtained by the method. From my own experience with the method I felt very much encouraged as to its reliability in giving concordant results, but a study of Tables IV and V emphasizes the fact that this method is sensitive (even more so than the neutral permanganate method) to changes in conditions governing its execution. The permanganate should be pure, the quantity exact, and the expression "below the boiling point" in the directions for the preliminary digestion should be changed to words more definite in meaning. In my own work I adopted as the temperature for the digestion a point just below that at which the contents of the flask would boil. The heat was so regulated as to bring them to this point in about ten minutes, after which they were kept not more than one or two degrees below boiling till the expiration of the thirty minutes, when the lights were turned up and the distillation completed in the time ordinarily required for a nitrogen distillation. No serious bumping or foaming of the contents of flasks was experienced in any of the work.

From the ease and rapidity with which this method can be executed, together with the individual results reported by the different analysts, I think it worthy of further trial. The most unsatisfactory feature connected with it seems to be its failure to place cotton-seed meal in the relative position indicated for it by vegetation experiments.

CONCLUSIONS.

While the results by the neutral permanganate method do not agree perfectly, they are sufficiently uniform to render the method of value in distinguishing between the different grades of nitrogenous material as determined by vegetation tests. This method places the availability of organic nitrogen at a much higher figure than is obtained in soil tests, but from a comparison of the results on all the materials that have been tested by it during the past four or five years, with the results obtained in pot experiments, there is a very satisfactory agreement in the relative position of their nitrogen availability.

The availability of nitrogen in materials of the first class is well up into the eighties and nineties, while garbage, leather, and such materials are well under 50, making quite a gap between them and rendering the detection of adulteration easier. Since about 40 per cent of the nitrogen in sample No. 6 is derived from garbage, it is apparent that a fertilizer, the determined availability of the nitrogen of which falls below 70 per cent, would be considered as of doubtful quality.

I hesitate to recommend the adoption of this method by the association in its present form, but, feeling that reliable results may be obtained by it when carefully executed, will give it to the association as follows:

Into a 400 cc Griffin beaker, low form, weigh an amount of sample containing, approximately, 0.075 grams of nitrogen (samples containing material that has been treated with acid should be washed on a 9 cm S. S. No. 595 filter to 200 cc and transferred, filter and all, to beaker); digest this with 125 cc of permanganate of potash solution (16 grams of pure KMnO_4 to 1,000 cc of water) in a steam or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water and keep closed with a cover glass, stirring twice at intervals of ten minutes with a glass

rod. At the expiration of the time remove from bath, add 100 cc of cold water and filter through a heavy 15 cm folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 cc. Dry and determine nitrogen in residue by Kjeldahl method.

RECOMMENDATIONS.

It is recommended—

- (1) That the neutral permanganate method be adopted as a provisional method.
- (2) That the alkaline permanganate method be further tested by the succeeding referee.
- (3) That a caution be inserted in the method for total nitrogen concerning the proper digestion bath of sulphuric acid with certain classes of material, or that the question be investigated by the referee.

The PRESIDENT. Are there any special papers relating to the determination of nitrogen, or any discussion of the paper presented?

Mr. Wiley is very desirous that some important work which has been done on cider by Mr. W. B. Alwood should be brought before the association, as several members of this body are interested in that line of work, and Mr. Alwood has kindly consented to make a brief statement in regard to it. I take this time to give him the opportunity of doing so.

ADDRESS OF MR. W. B. ALWOOD.

MR. PRESIDENT AND GENTLEMEN OF THE CHEMISTS ASSOCIATION: I am somewhat at a loss on my first appearance before a body of chemists, and especially such a dignified body of official chemists.

After hearing your president's address yesterday there is a question in my mind as to the specific gravity of a horticulturist or worker in economic botany. But I am happy to say that I have always been able to work in the most pleasant official relations with my brother chemists, and whenever I want to know anything about the constituent parts of any substance, I go to the chemist. In fact the whole data for the control of life problems I secure from the chemical laboratory.

Hence, after hearing your president's encouraging remarks on the importance to chemists of the work in the great field of life problems, it leads me to believe you will be interested in a work which I took in hand some years ago. This work was in a way forced upon me by its economic importance to fruit growers. And in the fewest words possible, it comprises methods of utilizing our unmerchutable fruit.

The fruit crop of the United States in the aggregate is enormous. Crop statistics generally give us in the case of apples only the merchantable crop, and in many cases only that which enters into foreign commerce. I have no adequate idea of the quantity of unmerchutable fruit produced in a crop year in the United States, but from statistics gathered in Virginia it is in my opinion safe to say that in a good crop year we have about 400,000 bushels of apples alone which fall below the best market grade.

I feel certain that anyone who has observed conditions in the great orcharding sections of our country will approve the statement that we simply do not know how much fruit of inferior grades goes to waste. And this is not true only of apples. How often does it happen that grapes are scarcely worth the expense of picking and marketing. I was told only this week of an instance where 400 tons of grapes were heaped up at a single New York cellar waiting to be used up in wine making. This implies that even first-class merchantable fruit may become a waste product because it can not be marketed.

I have said waste product not unadvisedly, because though we manufacture many products from our unmerchable and unmarketable fruits, we ordinarily waste the bulk of this material by reason of bad methods, or by making a product which is worse than waste.

To illustrate, the 400 tons of grapes mentioned above, were said to be piled up in mass, with the juice oozing out and dripping from them. This state of affairs indicates not only a loss of the must which thus runs away, but offers the best possible opportunity for all sorts of mal-germs to enter and begin their destructive work on the sugars of the fruit, and to contaminate the mass with flavors which can not afterwards be eliminated.

We heap up great quantities of inferior apples on the ground and then leave them to sweat and rot, thus destroying their intrinsic worth in sugar content, and allowing them to become seed beds for mal-organisms and to acquire in addition earthy flavors which can not be gotten rid of either in cider or other products made therefrom.

We also work inferior apples into sun-dried fruit in the South in great quantities. Two hundred tons of this product is often shipped in one year from our little depot at Christiansburg. This product returns as a gross price, 5 to 7 cents per bushel of apples to those who make it. Is this not an enormous waste of labor, let alone material? And it serves to put upon the market a product which injures the sale of better goods.

Now, the question arises, What shall we do with this refuse or unmerchable fruit? Canning, marmalade, etc., will consume an always increasing portion of it, but is there not room for a study of cider and vinegar making? This question has been thrust upon me in Virginia, and for the past eight years I have been working upon it with a view to reducing the methods and technique to a scientific statement.

First of all, I needed the chemical data on the composition of our fruits, and the changes which they undergo either in bad storage or improper fermentation processes. This data in American literature is very meager, though receiving increasing attention at your hands. Critical chemical data is absolutely necessary before I can, as a student of the fermentation processes, reach any conclusions of value.

Then also the question of the specific organisms which induce fermentation presents a study of the most vital importance, and it is on this phase of the subject that I have concentrated my part of the work. The American literature on this side of these questions is even more meager than on the chemical side. After seven years of work and experiment, I concluded that there was no better way to master the details of this subject than to go abroad and work it up in the foreign laboratories, where such leaders as Wortmann, Hansen, Linder, Fernbach, Kayser, and others had given much study to these subjects. This I was able to do through the generous assistance of Dr. Wiley, Chief of the Bureau of Chemistry, and through his continued assistance we have now established at Blacksburg an enological laboratory for the continuance of this work.

In this laboratory we have already brought together a representative collection of the alcoholic ferments, viz, the true yeasts of the group *Saccharomyces* which are used in Europe for the fermentation of some of the most noted wines and ciders, and also a collection of mal-ferments, and some of the acetic acid ferments. These latter fall correctly under the *Bacteriaceæ*, being known to botanists as *Bacterium aceti*. The use of the term *Mycoderma* to designate vinegar ferments is incorrect, though it occurs in the American literature up to the present date.

In addition to yeast races purchased abroad, I have also isolated a number of such races from material collected in Europe, and am now engaged on American material.

This seems to me a most promising field. While I do not for a moment believe the wide claims made in some quarters as to the efficacy of special yeast races in

producing with certainty the desired characteristics in a wine or cider, I know from my own work that these races when used in pure cultures in a proper manner will produce markedly different results, some desirable and others undesirable. Now, what we need to know is how to determine these facts with certainty, how to isolate and cultivate the desirable races in a commercial manner, and to devise a reliable technique for their practical use.

That color of the liquor, alcoholic strength, boquet, and flavor are affected by the organisms which produce the fermentation, is fully settled; but the "how" to surely bring about in a practical manner these desirable conditions and qualities is not settled by a long way.

We are however arriving at some knowledge of how the multiplicity of organisms, always present in any normal must, behave toward the same, and the conditions which favor or hinder their vital activities, and it is on these lines that the great field of life chemistry here opens up before the artisan who deals with these forces to produce a practical result.

I can not go into details in the few moments allotted to me, but I wish to repeat that we know enough at present to say that the key to good practical results appears to lie quite largely in the direction of pure cultures of strong yeast races which are capable of producing a sound fermentation, and taking possession of the must to the practical exclusion of mal-organisms. There is, of course, a great amount of detail as to cleanliness and temperature conditions and technique of handling the liquor. These will be treated in my reports to the Bureau of Chemistry.

The collections of yeast races and other organisms I have mentioned will, I believe, be put in shape for general distribution to specialists who may desire them. This matter rests with Dr. Wiley, and he should be consulted about it.

I am authorized to say that we need and earnestly request cooperation on the part of all the members of this association. The first desideratum is chemical data on all our fruit products, either fresh fruits, fruit must, wines, ciders, vinegars, etc. This part of the work in America presents a herculean task, and while Dr. Wiley's bureau is at work upon it, and Professor Davidson of our station is working on the chemistry of fruits and by-products of fruit, there is room for all, and we wish to compile and publish in this study as complete results as are attainable from all American sources.

Pardon a worker on the economic side for advising the chemists, but in your fruit analyses I wish to say that the next important ingredient to sugar and acid content is the tannin. This constituent of a fruit must is of the utmost importance in clarifying and assisting in other details of sound fermentation, hence it should always be determined.

Now, in conclusion, let me say this is not a small subject. It involves a study of the entire question of the preparation of alcohol in all its aspects, whether in beverages or liquors, for use in arts, manufactures, or science, and incidentally the preparation of all truly normal vinegar products.

I thank you for your indulgence.

THE PRESIDENT. I am sure we have all been glad to hear this report, and some, who have done work on the subject of cider vinegar, are more than ordinarily interested in these statements and will look forward with a great deal of hope and expectation to the future development of this work.

MR. ALWOOD. I did not touch vinegar. There also we are working out the races of the germ *Mycoderma aceti*, which is the true vinegar germ.

MR. EWELL. Mr. President, before you take up the regular pro-

gram I have a matter I would like to bring before the association. It is a suggestion for an amendment to the constitution. I bring it up inasmuch as a committee has been appointed for that purpose, and I would like to have you refer it to that committee. It refers to the question of membership in section 2. The part which I propose to strike out is as follows:

Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution.

This part of the section has never been adhered to, and as new members make inquiry concerning this point it rather complicates matters. I suggest the following in place of the words quoted above:

All persons eligible to membership shall become members *ex officio* and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials.

Referred to special committee on amendments.

The PRESIDENT. Next in order will be the report on insecticides.

Mr. HAYWOOD. Mr. President, the work on insecticides was divided into two parts, the arsenicals being given to the associate referee and the remainder of the subject to the referee. Professor Voorhees was not able to come to the meeting, but has sent his report. Mr. Street, however, was able to come down, and as he has been working with Professor Voorhees on this matter, I shall be very much obliged to him if he will read Professor Voorhees's report, to be followed by the report of the associate referee.

Mr. STREET. I have only slightly more familiarity with this work than Mr. Haywood, and have not even looked over the report, but I will read it.

INSECTICIDES AND FUNGICIDES.

By L. A. VOORHEES, *Referee*.

For reasons which were given at the time, the reports on insecticides and fungicides at the last two meetings of the Association of Official Agricultural Chemists embraced merely a consideration of proposed methods for various lines of work. This is the first year, therefore, in which any analytical work of a cooperative nature has been undertaken by the association. In order that the relative importance of the several lines of work, or, at least, the relative interest in the same might be discovered, requests, or invitations, to participate in all of the lines were sent to the members of the association in the shape of the following letter of inquiry:

DEAR SIR: Will you kindly inform me if you or any of your staff will cooperate in the work on insecticides this year; and if so, which of the following lines you are willing to take up:

Alkalinity in potash and soda lyes.

Arsenic in arsenicals.

Copper in copper carbonate.

Cyanogen in potassium cyanide.

Formaldehyde in formalin.

Nicotin in tobacco.

An early reply will be appreciated.

I remain, etc.

To this letter 48 replies were received, of which 26 were negative in their character and 22 affirmative. Many of the latter mentioned two or more lines in which they were interested, so that the number of individuals interested in each line was as follows:

Alkalies	6
Arsenic	14
Copper	9
Cyanogen	6
Formaldehyde	14
Nicotin	7

From this it will be noticed that equal interest was betrayed in arsenic and formaldehyde, and the interest in these two equaled the combined interest expressed in all the others.

Mr. J. K. Haywood, the associate referee, had very kindly consented to take complete charge of the work with the arsenic-bearing materials, an arrangement of which, in view of Mr. Haywood's experience in the study of the subject, the association can not fail to approve. To him the 14 names above alluded to were communicated, and from him you will hear the report of the work which has been done along that line. The writer himself took charge of the remaining lines.

As samples for the work in alkalies two commercial lyes were chosen, and with these were sent two samples of "whale-oil soap." The one was the product of James Good, of Philadelphia, and the other of Leggett and Brother, of New York City, both of whom kindly furnished an abundance of their product free of charge. To those who had expressed a wish to work with copper a sample of commercial copper carbonate was sent. Copper carbonate was sent also to each one to whom samples of paris green had been sent, with the request that copper determinations be made. As material to try the method proposed for cyanogen a sample of potassium cyanide was chosen. To those who desired to try the methods for formaldehyde a solution of formalin was sent, which was understood to run about 39 per cent of formaldehyde. To those who expressed a wish for the nicotin samples a tobacco extract and a tobacco powder were submitted. These samples were very generously supplied by Mr. R. G. Mewborne, chemist of the Kentucky Tobacco Product Company. With the samples were sent letters of instructions as to the methods and other particulars outlined in the proceedings of the last convention of the association.

Your referee regrets to report that the work which has been done along the lines coming under his eye is very meager. He has received but eight reports in all, of which two are on copper, two on cyanogen, and four on formaldehyde. These reports, while few in number, comprehend considerable work, and it has been deemed but fair to give them in full.

FORMALDEHYDE.

A. W. Ogden, of the Connecticut experiment station, writes as follows:

Inclosed you will find some results of determinations made on the referee's samples of formalin, and also on another sample of weaker formaldehyde, which we had on hand. In Table I are given the results by the Blank and Finkenbeiner method, and in Table II by the Legler, or ammonia method, as carried out in the following manner:

About $1\frac{1}{2}$ grams of formalin were weighed into a 3 or 4 ounce flask, containing an excess of standard ammonia, tightly stoppered, and shaken occasionally for sixteen hours, when the excess of ammonia was titrated with normal sulphuric acid, using one or two drops of a 0.5 per cent solution of corallin as indicator.

Some determinations were made by heating the mixture of formalin and ammonia in boiling water for one hour, cooling and titrating the excess of ammonia with sulphuric acid. By the latter method, I obtained 37.72, 38.15 and 38.63 per cent on your sample, and 20.38 and 20.94 on ours.

I see no advantage in heating, providing the mixture of formalin and ammonia is allowed to stand from twelve to sixteen hours in the cold. Where it stood two hours or less, as in determinations 1 and 2 in Table II, the results were low.

It took but a few minutes to make a determination by the Blank and Finkenbeiner method, while the results agree on the average with those by the ammonia method.

TABLE I.—*Referee's sample of formalin, 1901.*

[Blank and Finkenbeiner method.]

No.	Elapsed time between addition of H_2O_2 and titration.	Formalin.	2N-NaOH.	2N- H_2SO_4	2N-NaOH combined as CHO_2Na .	Formaldehyde (CH_2O).
	Minutes.	Grams.	cc.	cc.	cc.	Per cent.
1.....	3	1.3910	25.00	16.60	8.40	36.23
2.....	5	1.3357	25.00	16.50	8.50	38.18
3.....	5	1.1590	25.00	17.60	7.40	38.31
4.....	5	1.0753	25.00	18.15	6.85	38.22
5.....	5	1.1716	25.00	17.55	7.45	38.15
6.....	5	2.8859	30.00	12.20	17.80	37.06
7.....	8	3.0394	30.00	10.90	19.10	37.70
8.....	10	2.9854	30.00	11.30	18.70	37.58
9.....	10	2.1775	25.00	11.30	13.70	37.75
10.....	15	2.9620	25.00	6.40	18.60	37.68

RESULTS ON ANOTHER SAMPLE OF FORMALDEHYDE.

1.....	5	1.1825	25.00	20.90	4.10	20.80
2.....	5	1.3057	25.00	20.50	4.50	20.68
3.....	10	3.5635	25.00	12.80	12.20	20.54
4.....	10	.2740	25.00	24.00	1.00	21.90
5.....	10	.5423	25.00	23.05	1.95	21.58

TABLE II.—*Referee's sample formalin, 1901.*

[Legler, or ammonia, method.]

No.	Elapsed time between addition of NH_4OH and titration.	Formalin taken.	Standard ammonia (1 cc=0.004857 grams NH_3).	N. sulphuric acid (1 cc=0.017 grams NH_3).	Ammonia (NH_3) combined as $(\text{CH}_2)_6\text{N}_4$.	Formaldehyde (CH_2O).
	Hours.	Grams.	cc.	cc.	Gram.	Per cent.
1.....	$\frac{1}{2}$	1.3215	50.00	3.70	0.1799	36.03
2.....	2	1.3662	50.00	3.40	.1850	35.84
3.....	16	1.3280	50.00	3.10	.1901	37.89
4.....	16	1.3011	50.00	3.34	.1860	37.84
5.....	16	1.3189	50.00	3.20	.1884	37.81
6.....	16	1.3157	50.00	3.20	.1884	37.90
7.....	16	1.3204	50.00	3.20	.1884	37.77
8.....	16	1.3707	50.00	2.70	.1969	38.02
9.....	16	1.1900	50.00	4.20	.1714	38.13

RESULTS ON ANOTHER SAMPLE OF FORMALDEHYDE.

1.....	16	2.0775	50.00	4.35	0.1688	21.51
2.....	16	1.8970	50.00	5.18	.1547	21.58

The following results were obtained by Mr. R. W. Thatcher, of the Washington Experiment Station:

METHOD I.—Oxidation by H_2O_2 (Blank and Finkenbeiner)

Weight of formalin taken.	2 N-NaOH used.	Required for 3 grams.	CH ₂ O.
<i>Grams.</i>	<i>cc.</i>	<i>cc.</i>	<i>Per cent.</i>
3.3832	13.97	12.39	24.78
3.1838	13.60	12.76	25.52
3.1957	13.82	12.97	25.95
3.1407	13.80	13.14	26.28
3.1165	13.80	13.25	26.50
3.1663	14.36	13.57	27.14
2.9372	14.06	14.36	28.72
2.9288	14.07	14.41	28.82
Average . . .	-----	-----	26.72

METHOD II.—Weighing as hexamethylamine (Connecticut Station).

Weight of formalin taken.	Weight (CH ₃) ₆ N ₄ .
<i>Grams.</i>	{ Not determined, because compound was still losing weight after five weeks' drying in desiccator over H ₂ SO ₄ .
10.2400	
9.9870	

METHOD III.—Ammonia equivalent (Connecticut Station).

Weight of formalin taken.	N ₁₀ NH ₄ OH used.	CH ₂ O equivalent.	CH ₂ O.
<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Per cent.</i>
0.1841	0.02755	0.07273	39.49
.2458	.03587	.09470	38.50
.2403	.03590	.09477	39.41
Average . . .	-----	-----	39.13

METHOD IV.—Iodometric method.^a

Weight of formalin taken.	Iodine absorbed.	CH ₂ O equivalent.	CH ₂ O.
<i>Gram.</i>	<i>Gram.</i>	<i>Gram.</i>	<i>Per cent.</i>
0.1073	0.35682	0.04222	39.33
.0537	.17979	.02127	39.43
.0537	.17915	.02120	39.49
.1341	.44754	.05307	39.57
.1610	.53037	.06275	39.00
Average . . .	-----	-----	39.36

^a Allen's Common Organic Analysis, Vol. I, p. 220.

Method I gave entirely too low results. It is possible that 50 cc of my hydrogen peroxid was not sufficient to complete the oxidation. The fact that the larger the amount of formalin taken the lower was the per cent of CH_2O found might indicate this. Lack of material prevented my testing this point further. My observation would tend to cause doubt as to the reliability of the method as outlined at present.

Method II is far too slow for a practical method. Four weeks' drying in a desiccator, after evaporation at 75° , nearly to dryness, was insufficient to obtain constant weight.

Method III is easily carried out and gives fairly concordant results.

Method IV seems to me to be the most satisfactory one that I have tried. It gives very concordant results and is very quickly and easily carried out. It would be especially valuable for work with dilute solutions of formaldehyde.

Mr. A. E. Leach, of the food and drug laboratory of the Massachusetts State Board of Health, reports results by himself and by his assistant, Mr. Lythgoe, as follows:

Estimation of formaldehyde, Massachusetts State Board of Health.

	Weight of formalin taken.	2N NaOH required for weight taken.	Per cent formaldehyde CH_2O .
	Grams.	cc.	Per cent.
Leach, 1	3.2745	20.5550	37.66
Leach, 2	3.2365	20.3495	37.72
Lythgoe, 1	3.3220	20.8999	37.75
Lythgoe, 2	3.2284	20.5224	38.12

Our experience with the ammonia method is such that we have very little faith in its accuracy, referring especially to the method of evaporation. We find it almost impossible to get a constant weight, since loss of weight will continue as long as the hexamethylamine residue remains on the water-bath. I regret that we have not had opportunity to try the method of titrating the excess of ammonia with standard acid.

The method we prefer in this laboratory for estimation of formaldehyde is the iodometric method of Romijn.^a According to this method Mr. Lythgoe obtained a result of 38.27 per cent on your sample. A possible explanation of these low results may be the fact that the samples were kept in the laboratory more than two months before they were analyzed.

The following results were secured in the laboratory of the New Jersey Experiment Station, by the Blank and Finkenbeiner method. Mr. Street had a separate sample, which was bottled at the same time and in the same manner as those which were sent out to the other analysts, and a similar sample was retained for the use of your referee. The work was performed independently with different standardized solutions, which were afterwards compared with each other and found to agree. The samples also were exchanged.

^aZeits. anal. Chem., 1897, **36**, 18-24. Abstract Analyst, **22**, 221.

Estimation of formaldehyde (New Jersey station).

DETERMINATIONS BY MR. STREET.

Sample.	Weight of formalin taken.	2N NaOH required for weight taken.	Formaldehyde, CH ₂ O.
	Grams.	cc.	Per cent.
1. Street's sample	3.4459	22.49	39.16
2. Street's sample	2.5452	16.78	39.56
3. Street's sample	2.9057	19.18	39.60
4. Street's sample	2.8629	18.69	39.18
5. Street's sample	2.6992	17.76	39.48
6. Referee's sample	2.6458	17.31	39.25

DETERMINATIONS BY REFEREE.

1. Referee's sample	2.3645	15.42	39.13
2. Referee's sample	2.9774	19.50	39.30
3. Referee's sample	2.7040	17.68	39.23
4. Street's sample	3.1954	20.82	39.09
5. Street's sample	2.8566	18.59	39.05

COMMENTS.

A comparison of the results which have been reported shows that neither analyst had great difficulty in securing results by any one method, which agreed among themselves; the results by different methods were divergent, however. The lowest results by the hydrogen peroxid method are reported by Mr. Thatcher, but his results by the ammonia method and the iodometric method agree well with each other, and also with those secured by the hydrogen peroxid method by your referee and by Mr. Street. On the other hand, the results of Mr. Leach by the iodometric and hydrogen peroxid methods, and those of Mr. Ogden by the ammonia and hydrogen peroxid methods, are concordant, but all are about 1 per cent below the high results of Mr. Thatcher and those from New Jersey. If it is permissible here to use the axiom that things which are equal to the same thing are equal to each other, it would seem that no one of the methods is definitely proved to be worthless. The hydrogen peroxid of Mr. Thatcher may have been too weak, as he has suggested, and in the case of Mr. Leach and Mr. Ogden there may have been a depreciation in the sample. The method of weighing the portions taken for the determinations is also a matter of importance. The solution when exposed to the air gives off a strong odor of formaldehyde. That exposure occasions an undetermined but appreciable loss. In the work of your referee the portions for analysis were taken by weighing the formalin in a stoppered Schuster dropping bottle. Approximately 60 to 75 drops were then dropped directly into the standard sodium hydroxid, and the bottle was restoppered and weighed to show, by loss, the amount taken. Mr. Street, before this method was chosen, obtained results as low as 29.38 and no higher than 35.05 per cent. The strength and relation of the peroxid of hydrogen also has a bearing, for it was the observed fact with each of the analysts that the results varied inversely with the amount of formaldehyde to be oxidized. The time allowed to elapse before titration also seems to have had a bearing on the case.

The referee would like to add that the peroxid of hydrogen, which was used by Mr. Street and himself, was entirely free from acid, which is not the case with all samples of this product. Presence of acid would cause an apparent increase in the amount of formaldehyde present.

QUALITATIVE TESTS FOR FORMALDEHYDE.

With the samples of formalin, requests were sent to make tests of certain methods for its detection, qualitatively. Your reporter had no intention of trespassing on the field of the referee on food adulteration, or of his associate, who had the matter of preservatives in hand, and his apology is here tendered. The few results secured are presented, however, in order to deal fairly with the analysts who so kindly carried out the tests.

Mr. A. W. Ogden, of the Connecticut station, reports as follows:

A solution containing 1 part formaldehyde to 1,000 parts water was made by diluting 1.3 grams of the referee's sample of formalin to 500 cc with distilled water. To 10 cc of this solution were added 990 cc of milk, making a mixture containing 1 part formaldehyde to 100,000 parts milk. Good tests were obtained on this milk, using the following methods.

Hydrochloric acid test.^a

Ten cubic centimeters of milk were shaken in a test tube with 5 cc of strong hydrochloric acid containing 1 drop of a ferric chlorid solution and gently heated, never to boiling, with constant agitation. The presence of formaldehyde was indicated by a purple color.

Sulphuric acid test.

To 5 cc of milk in a test tube an equal amount of strong sulphuric acid, containing a trace of ferric chlorid, was carefully added so the two did not mix; a violet or purple ring at the point of contact denoted the presence of formaldehyde. This test appeared more delicate when the milk was mixed with three or four times its volume of water, and three or four drops run carefully, to avoid charring, into a test tube containing 5 cc of the strong sulphuric acid mixture and 3 drops of ferric chlorid to 100 cc of acid.

Rimini test.^b

One hundred cubic centimeters of milk, with one drop of a 50 per cent citric acid solution, and a piece of paraffin, were distilled until 20 cc had come over. To 15 cc of the distillate were added 1 cc of a solution of strong phenylhydrazine hydrochlorid (4:100), 4 drops of a freshly prepared solution of sodium nitro-prussid and 4 or 5 drops of a 50 per cent sodium hydroxid solution. Formaldehyde was indicated by a green coloration, which changed to red on standing. Fifteen cubic centimeters of distillate from a sample of milk, containing 1 part formaldehyde in 20,000 parts, gave with Rimini's test a beautiful blue coloration, which soon changed to green and finally to red.

Mr. R. W. Thatcher reported as follows:

I prepared solutions containing 1 to 10,000, 1 to 20,000, etc., up to 1 to 1,000,000 parts of formaldehyde and tested by the various reagents indicated below. In each case after applying the reagent to 10 cc of the solution, the mixture was allowed to stand fifteen minutes and at the end of that time I noted first, the limit to which the color change was sufficiently striking to afford what I would deem a reliable evidence of the presence of formaldehyde, and second, the limit to which any color change could be detected by comparing with a blank tube containing no formaldehyde. The following results were observed:

Qualitative determinations of formaldehyde with various reagents.

Author.	Reagent.	Limit of reliability.	Limit of sensibility.
Hehner.....	H ₂ SO ₄ + trace of Fe Cl ₃	No change	in any case.
Schiff.....	10 cc of fuchsin decolorized by SO ₂	1 to 20,000	1 to 60,000
Do.....	Same after standing 1 hour.....	1 to 100,000	1 to 400,000
	Phenol pour into H ₂ SO ₄	1 to 100,000	1 to 400,000
Lebbin.....	1 cc 5 per cent solution of resorcin.....	1 to 100,000	1 to 600,000
	1 cc 5 per cent solution of NaOH.....		
Vanini.....	5 drops 1 per cent solution of phloroglucin.....	1 to 20,000	1 to 1,000,000
	5 cc 4 per cent solution of NaOH.....		

^a Conn. Agr. Expt. Sta. Rept., 1900, p. 127.

^b Anal. di Farinacol, 1898, 97 (Abstract Zeitschr. Unters. Nahr. Genuss, 1, 858.)

I would recommend that the Hehner test be taken from the list of official methods, as it is very unsatisfactory and unreliable with even moderately dilute solutions. The Schiff's reagent gives the most striking and most easily perceptible color change, but is not sensitive to very small quantities of formaldehyde, unless the mixture is allowed to stand for one hour at least. I found the phloroglucin test, as recommended by Vanini, to be the most satisfactory of any that was tried, but the pink color is not very pronounced and not so easily seen as some of the others.

Mr. A. E. Leach reported as follows:

We have had occasion many times to try the qualitative tests for formaldehyde. In our regular milk work we test all samples for preservatives during the months of June to September, inclusive, amounting to about 20 samples per day. The preliminary test that we have adopted as being by far the most satisfactory, is that of heating below boiling with hydrochloric acid and ferric chlorid.^a This is much more delicate than the sulphuric acid method, in which the charring effect of the acid is apt to obscure the test when only minute quantities are present. We have satisfactorily used this test for four years, and find it delicate to one part formaldehyde in 300,000 parts milk.^b

Mr. J. P. Street reported qualitative tests as follows:

To 620 cc of water were added 1.63 grams of formalin, equaling 1 part of formalin in 1,000. Ten cubic centimeters of this diluted formalin were added to 490 cc of milk, equaling 1 part of formalin in 50,000. A portion of this was still further diluted to 1 part in 100,000 and one part in 200,000.

Test 1.—Ten cubic centimeters milk were shaken with 5 cc hydrochloric acid, containing a drop of ferric chlorid and heated below boiling for three to five minutes. A violet color showed the presence of formalin.

Test 2.—Ten cubic centimeters of milk were taken and an equal volume of sulphuric acid added, containing a trace of ferric chlorid. The formalin was shown by the violet ring at the junction of the two liquids.

Test 3.—Five grams of coarsely powdered potassium sulphate were weighed into a 100 cc flask, 5 cc milk added, and 10 cc sulphuric acid carefully poured down the sides of the flask. A bright violet color was formed.

All of these tests gave a clear, characteristic reaction in each of the three diluted solutions. Test 1, though very apparent, gave a little weaker reaction than the other two tests in the solution, 1 to 200,000.

COPPER DETERMINATIONS.

Two reports on copper have been received, the one from Mr. R. W. Thatcher, of Washington, and the other from Mr. Charles A. Peters, of Idaho. Mr. Thatcher's work was planned "to show the effect of varying quantities and kinds of ammonium salts, or free ammonia, upon the end reaction when titrating with potassium cyanid," and his "observations seem to show conclusively that the use of sodium carbonate as the neutralizing agent, with a small amount of ammonium hydrate added to clear the solution and give the deep purple color, is the only reliable method of procedure." His methods were as follows:

Method I.—Dissolve in a measured quantity of ammonium hydrate (sp. gr. 0.96), and titrate directly.

Method II.—Dissolve in nitric acid, add measured excess ammonium hydrate, and titrate.

Method III.—Dissolve in hydrochloric acid, add measured excess ammonium hydrate, and titrate.

Method IV.—Dissolve in nitric acid, neutralize with sodium carbonate solution, add 1 cc ammonium hydrate (0.96), and titrate.

Method V.—Dissolve in hydrochloric acid—proceed as in IV.

^aThirty-first An. Rep. Mass. State Board of Health, 1899, p. 606.

^bThe limit of delicacy was erroneously stated as 1:100,000 in our 1899 report.

Mr. Thatcher's results are as follows:

Determination of copper.

	Copper carbonate.			Paris green.		
	a.	b.	Average.	a.	b.	Average.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Method I	Not applicable.			22.28	22.34	22.31
Method II	56.00	56.14	56.07	24.32	24.00	24.16
Method III	53.88	53.64	53.76	23.97	23.63	23.80
Method IV	57.72	57.64	57.68	24.77	24.77	24.77
Method V	57.82	57.72	57.77	24.66	24.77	24.71
Moisture *	1.73	1.75	1.74	1.04	1.04	1.04

* Loss on drying eight hours at 105° to 110°.

Mr. Peters reports that Mr. H. T. Beans, who conducted the determinations, used the following procedure:

The paris green was dissolved in nitric acid, sodium hydroxid was added to the solution until a slight precipitate formed, 10 cc of a saturated solution of sodium carbonate were added, followed by 1 cc of ammonia (sp. gr. 0.90) the volume was made up to 100 cc, and the solution finally was titrated with potassium cyanid. The potassium cyanid was standardized against a copper-nitrate solution, the copper contents of which were determined electrolytically. The copper carbonate sent by referee contains considerable copper oxid. A portion of the copper carbonate furnished was ground, triturated, and the homogeneous sample thus obtained was used in this work.

Mr. Beans's results were as follows:

	Copper carbonate.			Paris green.		
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Copper	55.56	55.63	55.60	24.81	24.59	24.70
Moisture	1.54	1.75	1.65	0.92	0.83	0.88

* These moisture determinations were made on the original sample of copper carbonate and not on the ground sample upon which the other determinations were made.

POTASSIUM CYANID.

Mr. C. L. Penny reports results with potassium cyanid, as follows:

Your letter of instruction on determining cyanogen, together with a sample of potassium cyanid has been received. I have to report the following results of analysis by the Liebig method, as stated by you. I weighed out 10 grams in duplicate from the center of the bottle, and made up to 1,000 cc. For each determination I used 50 cc equivalent to 0.5 grams. In one case the decinormal silver solution was 36.9 cc, in the other 37.0, the mean 36.95 cc. Calculating from the mean I find the cyanogen (CN) to be 38.49 per cent, and hydrocyanic acid (HCN) to be 39.97 per cent. A perfectly pure and water-free salt would call for CN=39.94 per cent.

The atomic weights used are K=39.15, CN=26.04, H=1.01. The method seems to me to be easy of execution, and to leave little to be desired. In a supplementary trial I added sodium chlorid, in amount equivalent to about one-half of the cyanid, and found the chlorid had no effect whatever on the result.

I tried chromate of potash as an indicator, and got 29.70 cc and 29.75 cc of silver for 0.2 gram of salt. This latter method gave 38.70 per cent cyanogen, as compared with 38.49 per cent by the Liebig method. The chromate method has the great disadvantage of showing chlorid as cyanid.

I should consider the Liebig method, as stated by you, admirable in every way, and leaving nothing to be desired.

Mr. Street, of the New Jersey Experiment Station, reported 39.29, 39.34, and 39.30 per cent of cyanogen in another sample of this material, using the same method. It would be well to state that the samples of potassium cyanid which were sent out were purchased already put up in 2-ounce bottles, and a difference in samples may account for this difference in results, which in view of this fact should not be considered so very great.

REMARKS.

Your referee on the subject of insecticides and fungicides regrets that his report can not be made of more interest by having the results of more work to report, and trusts that the associate referee, Mr. Haywood, will be able to counterbalance the deficiency by submitting a report of considerable work on the arsenicals.

The work here reported is so meager that your referee would refrain from making any recommendations based thereon. He would, however, venture to call the attention of the association to the following considerations:

First.—The detection and determination of formaldehyde does not properly belong to the referee on agricultural insecticides and fungicides. Except for fumigating closets, etc., or for use in the laboratory, I do not believe it is employed at all extensively.

Second.—The content of cyanogen in a sample of cyanid of potash is of interest, but for fumigating a greenhouse or similar purposes I believe a knowledge of the impurities and the character of the gases which they supply would be of greater value.

Third.—The purity of the lyes on the market for the home making of insecticide soap is of importance, but when it comes to the ready-made soaps themselves, there is much in the regular soap analysis that is of no interest to the entomologist.

From these considerations it would seem that much of minor importance might be eliminated from the work laid down by your referee which would result in lightening the labor of his successor. The recommendation is therefore made that the methods be continued with this in mind.

The PRESIDENT. We will now have the other portion of the report on insecticides.

Mr. HAYWOOD. This is the report on arsenicals from the associate referee:

REPORT ON THE ARSENICAL INSECTICIDES.

By J. K. HAYWOOD, *Associate Referee*.

At the request of Mr. Voorhees, the referee on insecticides, the associate referee took charge of the work on the arsenicals. Thirteen chemists signified their desire to take part in this branch of the work. The following directions were sent to each, accompanied by materials for analysis:

DEAR SIR: You recently informed Professor Voorhees that you would collaborate in the work on insecticides. He has placed the work on arsenicals in my hands.

Inclosed you will find materials and methods for the determination of the two forms of arsenious acid in paris green. Will you kindly report all results as per cent and let me have them by October 15, if possible? Mix the sample of paris green well before analysis.

Very truly, yours,

J. K. HAYWOOD, *Associate Referee*.

METHODS OF ANALYSIS FOR INSECTICIDES.

PARIS GREEN.

MOISTURE.

Dry 1 to 2 grams for 8 to 10 hours at 105° to 110° C., and calculate loss as moisture. (In duplicate.)

TOTAL ARSENIOS OXID.

Method I.

Solutions required:

Starch solution.—To prepare the starch solution, boil 2 grams of starch with 200 cc of water for about 5 minutes.

Iodin solution.—To prepare the standard iodine solution, dissolve 12.7 grams of powdered iodine in about 250 cc of water to which has added 18 to 25 grams of c. p. potassium iodide, and make the whole up to a volume of 1 liter. To standardize this solution weigh out 1 gram of the inclosed dry c. p. arsenious oxide; transfer to a 250 cc flask by means of about 100 cc of a solution containing 2 grams of sodium hydrate in each 100 cc, and boil until all arsenious oxide goes in solution; cool; make to a volume of 250 cc and use 50 cc for analysis.

This 50 cc portion is concentrated by boiling in a 250 cc flask to half its volume and allowed to cool to 80° C. An equal volume of concentrated hydrochloric acid is now added, accompanied by 3 grams of potassium iodide, mixed, and the whole allowed to stand for 10 minutes (to reduce the arsenic oxide, formed on boiling an alkaline arsenite, to arsenious oxide). The brown solution is then diluted with cold water and an approximately N/10 solution of sodium thiosulphate added, drop by drop, until the solution becomes exactly colorless. (This end point is easy to read without the aid of starch.) This solution is then made slightly alkaline with dry sodium carbonate (using a drop of methyl orange to read the change), and made slightly acid with hydrochloric acid, *taking care that all lumps of sodium carbonate on the bottom are acted on by the hydrochloric acid.* Sodium bicarbonate is now added in excess and the solution of iodine run in, drop by drop, using starch solution to read the end reaction. (Sometimes the solution gets dark toward the end of the titration. This must not be confused with the final dark blue color given by the iodine and starch.)

From the number of cc of iodine solution used and the weight of arsenious oxide taken, the value of each cc of iodine in arsenious oxide can be determined.

Method (Smith modified by Haywood).—Two grams of paris green are weighed out and transferred to a 250 cc flask and about 100 cc of water and 2 grams of sodium hydrate added. This solution is boiled for 5 to 10 minutes, or until all of the green particles have changed to red cuprous oxide. It is then cooled to room temperature and the volume made to 250 cc. The well-shaken liquid is filtered through a dry filter and 50 cc taken for analysis. The analysis is carried out from this point forward the same as when we standardize the iodine solution. (In duplicate.)

Method II.

Solutions.—The solutions required are the same as above, with the addition of a solution containing 2 to 3 grams of sodium potassium tartrate in 50 cc of water.

Method (Avery-Beans).—Sample the paris green (as one would an ore for assaying) down to about 1 gram. Pulverize this small sample in an agate mortar and weigh out 0.2 to 0.3 gram in a beaker of, say, 300 cc capacity. Add 25 cc of water, and to the green suspended in the water add, with constant stirring, concentrated hydrochloric acid till solution is just effected. Six drops are usually sufficient. Now add to the acid solution sodium carbonate solution till a slight permanent precipitate is formed. Dissolve this precipitate by adding 2 to 3 grams of sodium potassium tartrate in solution. Now dilute to about 200 cc, add solid sodium bicarbonate and starch solution, and titrate with iodine in the usual way. (In duplicate.)

Method III.

(By precipitation with hydrogen sulphide and weighing as arsenious sulphide.) Dissolve with hydrochloric acid, filter, heat the solution in a flask to 70° C., and conduct hydrogen sulphide through as long as precipitation takes place. The precipitate formed is a mixture of sulphur, arsenious sulphide, and copper sulphide.

Spread out the filter in a porcelain dish with watch glass cover, and digest over water bath with sodium sulphid solution. Decant and repeat digestion two or three times. Finally, add some water, filter off with clear solution, and wash the residue with hydrogen sulphid water.

The sodium sulphid solution is warmed and made acid with hydrochloric acid, which reprecipitates the arsenious sulphid and a large amount of free sulphur; filter and wash. Extract the still moist precipitate on the filter with ammonia, wash the residual sulphur, reprecipitate the solution with hydrochloric acid without heat, filter in a Gooch crucible, dry, extract the remaining free sulphur with carbon disulphid, dry at 100°C ., and weigh. Repeat extraction and drying until constant weight is secured. From the arsenious sulphid compute arsenious oxid. (In duplicate.)

Method IV.

(C. A. Goessmann.)

Weigh out 1 gram of the material in a beaker, add 50 cc of water and 5 grams of potassium hydroxid and digest on boiling water bath for half an hour. Filter and wash the precipitated suboxid of copper, make the filtrate acid with hydrochloric acid, and change the arsenious acid to arsenic acid by the addition of small quantities of chlorate of potash at frequent intervals and gentle heat. Drive off the excess of chlorin by repeated evaporations, make alkaline with ammonia, and precipitate with magnesia mixture. Let stand at least two hours, filter through a Gooch crucible, dry and burn, using in so doing the precautions mentioned in Fresenius. Cool and weigh as magnesium pyro-arsenate. (In duplicate.)

SOLUBLE ARSENIUS OXID.

Method I.

Solutions.—A solution of starch and the standard iodine solution prepared as above; also a solution of sodium acetate containing 12.5 grams in each 25 cc.

Method—(Avery-Beans).—Digest over the flame 1 gram of paris green for about 5 minutes with 25 cc of a solution of sodium acetate containing 12.5 grams of the crystallized salt. The solution is then cooled, made up to 100 cc and 50 cc filtered off and titrated with standard iodine in the usual way. (In duplicate.)

Method II.

Solutions.—A solution of starch and the standard iodine solution prepared as above.

Method—(Haywood).—One-half gram of paris green is treated with 500 cc of water (previously boiled to get rid of CO_2 and again cooled to room temperature) in a large flask. The flask is stoppered and shaken 8 or 10 times each day for 10 days. At the end of this time the solution is filtered off through a dry filter. Two hundred cc of this are treated with sodium bicarbonate and titrated with iodine. This will give the percentage of arsenious oxid dissolved.

Another 200 cc of the solution is treated with 5 cc of concentrated hydrochloric acid, heated to 70°C ., and a current of H_2S passed through until all arsenic and copper are precipitated. It is then filtered and washed quickly with hot water. The filter and its contents are burned with powdered sulphur and weighed in a porcelain crucible. In this way a mixture of CuO and Cu_2S is obtained. To calculate the percentage of CuO dissolved, consider the residue as entirely made up of CuO . This will be correct, since the per cent of copper in CuO and Cu_2S is the same. (In duplicate.)

Please send both the figures for arsenious oxid and cuprous oxid dissolved by Method II and the reporter will calculate the CuO to arsenious oxid and subtract that amount from the total amount found.

Before going into the results obtained I will give a brief résumé of what has been done on the determination of soluble arsenious oxid in paris green, to at least explain why the methods as given above were chosen.

Up to about one year ago there had been no methods worked out for the determination of free arsenious oxid in paris green. Some chemists would allow the green to stand in contact with water for two to four hours and titrate the resulting solution with standard iodine; some would allow the green to stand in contact with water for twenty-four to forty-eight hours and titrate the resulting fluid; some would wash the

green on a filter paper with a liter or two of water and titrate the resulting solution; some would extract the green with warm water, etc. By these methods many and diverse results were obtained on the same sample of paris green.

The first work that I can find on this subject was by Haywood and appeared in the *Journal of the American Chemical Society* in September, 1900. It was found during the course of the investigation there recorded that warm water could not be used to extract the soluble arsenious oxid since it broke up the paris green, also that it was not practicable to extract the soluble arsenious oxid by washing on a filter paper, since the wash liquor was still gaining in arsenious oxid, even after 2,000 cc had been used. It was also found that the soluble arsenious oxid went into solution much more slowly than had been known or anticipated, so that all had not gone into solution even at the end of a week, but seemed to require from ten to twelve days. A method was finally proposed based on these facts. It is as follows:

Suspend one-half gram of paris green in 500 cc of water in a stoppered flask. Shake occasionally each day, and at the end of ten to twelve days filter off an aliquot portion and titrate with iodine solution for soluble arsenious oxid.

A month later an appendix appeared to this paper, in which the author further stated that copper oxid also went into solution to some extent during the extraction with cold water. The author was inclined to think that this was due to a breaking up of the paris green, and so assumed (no work having been done to prove the contrary) that the copper oxid and arsenious oxid went into solution in water in the same proportion in which they were present in the paris green, and consequently a nearer figure to the true per cent of soluble arsenious oxid could be obtained by correcting the total quantity by an amount corresponding to the copper oxid in solution.

During the same month, October, 1900, another paper on the soluble arsenious oxid in paris green, by Hilgard, appeared in the *Journal of the American Chemical Society*. With the former writer he agreed that warm water could not be used to extract the soluble arsenious oxid from paris green, but, unlike the former worker, he seemed to think that the copper in solution in the water came from verdigris, perhaps already present in the green, and that, after washing the green for a while, although arsenious oxid still came through in the wash water, copper oxid failed to do so, thus showing that the paris green did not break up when in contact with water, and that, consequently, a correction could not be applied to the soluble arsenious oxid based on the amount of copper oxid found in solution. He advised extracting 1 gram with 1,000 cc of water for twenty-four hours, at least, and using the resulting figure, obtained after titration, for free arsenious oxid.

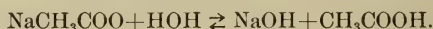
In February, 1901, a paper by Avery and Beans appeared in the *Journal of the American Chemical Society* on this subject. They found, during the course of their investigation, that water does break up paris green to a great extent (depending on the fineness of the material), but that the amounts of copper and arsenious oxid going into solution are not in the same proportion as that in which these substances are present in the original green. Although they were of the opinion that approximate results could be obtained by suspending 1 gram of paris green in 1,000 cc of water for a week, they said that this method was purely arbitrary, and therefore advocated extracting the paris green with a boiling solution of sodium acetate, which compound not only renders the free arsenious oxid present more soluble than it would be in pure water, but retards the dissociation of the paris green, thus causing it to be broken up to a much less extent. They also found that water saturated with CO_2 dissolved about one-half the arsenious oxid from a sample of paris green during the course of a week.

In this connection it may be of interest to show some figures that we have obtained at the Bureau of Chemistry, Department of Agriculture, comparing the water extraction and sodium acetate extraction methods, and to call attention to certain facts brought out by them.

Extraction of arsenious oxid by water and by sodium acetate.

Number.	Arsenious oxid solu- ble in water.	Copper oxid solu- ble in water.	Arsenious oxid solu- ble in sodium ace- tate solu- tion.	Number.	Arsenious oxid solu- ble in water.	Copper oxid solu- ble in water.	Arsenious oxid solu- ble in sodium ace- tate solu- tion.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	9.30	1.90	.88	1.....	2.16	.90	1.08
2.....	8.81	1.45	1.17	2.....	4.11	.65	1.17
3.....	6.37	.65	1.76	3.....	4.32	.55	1.17
4.....	6.97	1.65	1.08	4.....	4.41	1.05	1.08
5.....	5.52	.95	1.17	5.....	5.39	.90	.88
6.....	10.53	.60	8.98	6.....	2.93		.88

From this table it will be seen that the figures for arsenious oxid by water extraction are much higher than those by sodium acetate extraction, even when the water extraction figures are corrected by subtracting an amount of arsenious oxid corresponding to the amount of copper oxid in solution. But we would like to call your attention to another fact. The first six of these greens are the very coarsest that could be found among a group of forty-seven and the second six the finest. It therefore appears that the water extracted in every case more arsenious oxid from coarse paris greens than from the fine ones; yet Avery and Beans found that the finer the green the more arsenious oxid extracted in a given length of time. We agree with Avery and Beans that water does break up paris green, and that for any certain green the finer the green the more it will be broken up by water in a given length of time, since more surface is exposed to the action of the water. Yet these figures would seem to show the contrary. This apparent discrepancy is, perhaps, explained when we consider the following fact. The coarser paris greens are considered in most cases not to be made as well as the finer ones. Might it not be the case, then, that such greens have not been boiled long enough in acetic acid, and that a certain part of them is in a very loose combination, which combination is readily broken up on treatment with water? On the other hand, when such samples of paris green are treated with sodium acetate we would have the following conditions. Paris green would be suspended in a solution containing a large amount of sodium acetate, which, as is well known, is hydrolyzed to a great extent in water solution according to the following scheme:



Might not the acetic acid act on the sample in question and cause the arsenious oxid to form a more stable compound, so that nearly all of the arsenious oxid going into solution would be that not combined in any way with the other constituents?

Since a small amount of NaOH also exists in the above solution we would naturally expect that it would act on the paris green to some extent, and set a little arsenious acid free over and above that present in the free state, along with some copper. Such an action very likely does take place, since small amounts of copper are always found present in solution on extracting the green with sodium acetate. That such an action would be small as compared with the "combining" action of the acetic acid in solution seems to be borne out by the above figures.

Looking at the matter in this light, it would seem that even though water extraction does not give the exact amount of free arsenious oxid in the green, it at least gives us some idea of its stability and is consequently of value.

For these reasons your associate referee has chosen the sodium acetate method as the best one now at our command to determine the actual soluble arsenious oxid in

paris green, and the water extraction method as the one which shows to some extent the stability of the green in question and gives some idea of what its action would be in actual orchard practice.

Following are the results obtained by five chemists. The other eight were not able, because of press of work, to send in any figures at all.

Estimation of total and soluble arsenic.

Analyst.	Moisture.	Total arsenious oxid.				Soluble arsenious oxid.		Soluble copper oxid.
		Method I.	Method II.	Method III.	Method IV.	Method I.	Method II.	Method II.
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
H. T. Beans, Moscow, Idaho.....	0.92	56.29	56.63			0.78	0.86	0.30
	.83	56.43	56.71			.86	.96	.40
			56.63					
R. W. Thatcher, Pullman, Wash	1.04	56.12	56.16			.55	2.94	
	1.04	56.03	56.29			.52	2.88	
J. K. Haywood, Washington, D. C. ...	.91	56.68	56.84		* 56.10	.78	2.08	.40
	.88	56.80	56.82		56.00	.81	2.69	.55
		56.80						
G. W. Cavanaugh, Ithaca, N. Y.		55.88	57.16		* 56.30			
		56.00	57.16		56.35			
		56.13						
H. B. Slade, Lincoln, Nebr.	Trace.	56.69	55.98		56.10	.42		
		56.81	56.31		55.97	.42		

* Corrected as directed by Fresenius for the solubility of the magnesium ammonium arsenate precipitate in the mother liquor.

REMARKS.

H. T. Beans.—(1) In Method IV for total arsenious oxid considerable amounts of iron from the paris green and silica from the glassware interfered with the determination.

(2) An objection is made to the standardization of the iodine solution for the tartrate method (Avery-Beans) by the process outlined for the Smith-Haywood method, unless it can be shown that there is an appreciable oxidation of arsenic during its solution as carried out by the ordinary method of preparing alkaline arsenite solution. The authors (Avery-Beans) prefer to standardize the iodine solution in the regular way, i. e., by the use of a solution containing 4.95 grams of arsenious oxid in a liter of water.

R. W. Thatcher.—I did not attempt the estimation of arsenic as sulphid, as my laboratory is not equipped for combustion in hydrogen, and furthermore I believe that the method is practically obsolete as far as being a practical method for technical use. I did attempt the determination by the Goessmann method. but upon making the solution alkaline for precipitation with magnesia mixture a very perceptible quantity of aluminum hydroxid appeared, probably derived from the dish in which the paris green was digested with caustic alkali. Furthermore, the solution to which the magnesia mixture was added gave a continued deposition of crystalline matter for several days. I finally became disgusted and threw away the whole of the precipitated matter. I am very strongly of the opinion that the gravimetric methods as at present outlined are entirely worthless.

J. K. Haywood.—I was not able to get reliable results by Method III for total arsenious oxid. Nearly always the copper sulphid would run through the filter after being separated from the arsenious sulphid by sodium sulphid. Even when this did not happen the final results on arsenious sulphid were very variable.

Geo. W. Cavanaugh.—I omitted Method III for total arsenious oxid, since from previous experience I found that I could not get reliable results with it.

H. B. Slade.—The determination of arsenic as arsenious sulphid was made, but the results were so divergent, owing to the large amount of sulphur present, and the time taken in washing out the sulphur led me to omit the completion of this method. The Smith-Haywood method (I) for total arsenic apparently gives high results from lack of sharp end reaction with thio-sulphate in the preliminary treatment. The iodine is thus titrated against it and the percentage increased by this error. The same error, if present, is involved in the standardization of the iodine solution. In each case in the determinations reported 19.5 cc of thio-sulphate solution were necessary to take up the iodine set free in this method. The error, then, appears constant.

RECOMMENDATIONS.

Your associate referee would recommend that Methods I and II be adopted provisionally and tested again next year, and that both methods for soluble arsenious acid be given another trial.

The PRESIDENT. Are there any special papers to be presented on the subject relating to the analyses of insecticides? Are there any remarks or is there any discussion of the report?

Mr. ALWOOD. I would like to know whether there is any method of treating paris green so as to get rid of the soluble arsenious acid and to prevent the paris green itself from breaking up? Is there any method by which the horticulturist or farmer could treat the paris green?

Mr. HAYWOOD. It has been thought for a long time that if paris greens were treated with calcium hydroxid in solution it would render the free arsenic more insoluble, or at any rate improve them. Personally I believe that calcium hydroxid may break up the paris green to some extent, and really renders the total arsenic more soluble, but that this arsenic is not more soluble as arsenious acid but as calcium arsenite, which does not seem to injure the foliage as much as arsenious acid. Probably more work will have to be done on this before we can tell what the action of calcium hydroxid is.

Mr. ALWOOD. Some years ago we did some work in our laboratory with reference to the soluble arsenic in London purple, and, whatever the explanation was, it was very evident that the addition of quicklime or calcium hydroxid to the London purple diminished very noticeably—not entirely, but below the danger point—the solubility of the arsenic.

(The president asked Mr. Wheeler to take the chair, and then spoke from the floor.)

Mr. VAN SLYKE. I want to make just one or two statements in regard to the subject of soluble arsenic in water. Now it seems to me that it is desirable, in determining the amount of soluble material in paris green, to keep in mind the specific reason for which we want to make the determination. It is known that arsenic in soluble form injures foliage when it is present beyond certain quantities, and it seems to me that in our methods we need to keep in mind this partic-

ular thing. This is a question of scientific interest, and it is desirable to determine what the specific effect of sodium acetate is and what the specific effect of the water is on the paris green. But when it comes to the practical application of our results in horticultural work, it seems to me that we ought to adopt the method which will apply to the question of the determination of the danger point in the application of the arsenic to foliage. In my own judgment it appears that we should approximate somewhat the conditions which maintain in horticultural operations. Ordinarily the paris green is mixed with the water not very long before it is applied, probably not more than a few hours at the most. It is possible, but I do not think it is a common custom, for horticulturists to mix the paris green and let it stand indefinitely before using. Now, if the paris green is in contact with the water only a few minutes or a few hours before application, what is the use of making a determination of the amount of arsenic that is soluble in sodium acetate or in water on standing a week, more or less? In the absence of any better guide in our own laboratory, where we are obliged to look after the paris green of the State by way of inspection, I have provisionally adopted the method of putting the water on paris green at the rate of 10 parts of water to 1 of paris green and shaking occasionally and letting it stand in contact for twenty-four hours. Now, it is safe to say that if that treatment fails to develop an amount of soluble arsenic that will be sufficient, in the opinion of horticulturists, to injure the foliage, then it is perfectly safe to pass that paris green as all right. In working over the samples that are present in the market of New York State, I believe there was only one that failed to respond to this test; that is, there was only one sample that appeared to contain sufficient soluble arsenic to make it possible that the foliage might be injured. On the other hand, where we let it stand ten days or two weeks the amount of soluble arsenic was so large that scarcely any of the samples were of a kind to be permitted in horticultural operations. So I say that it seems to me that while it is desirable to study the actual chemical changes that take place, we want to keep in mind the application, and that we want, if possible, to approximate conditions in our methods that will enable us as nearly as possible to tell how much soluble arsenic is present in the paris green that in the hands of the horticulturist, under the methods used by him, is likely to do harm.

Mr. HAYWOOD. I would like to say in this connection that in determining the soluble arsenic in paris green, although you may get certain results by dissolving for twenty-four hours, or overnight, really the result which gives you more closely the effect the paris green would have in actual orchard practice is to let it stay a good length of time in contact with water, then you will have some idea of its stabil-

ity. It depends a great deal on the stability of the paris green, it appears to me, whether it will scorch the foliage or not, because an unstable sample of paris green, under adverse conditions, with a great deal of moisture and perhaps quite a little carbon dioxid coming in contact with it, might scorch the foliage. So it appears to me that what really is needed is to understand paris green from the standpoint of its stability even more than from its sodium-acetate-extraction standpoint. Even the free arsenic that is present hardly begins to pass into solution in twenty-four hours, leaving out of consideration the breaking up of the paris green.

Mr. VAN SLYKE. I would like to ask Mr. Haywood if he has any information which shows whether, after the paris green is applied to the foliage, any continuous change goes on? I have not been able to find any information published on that point.

Mr. HAYWOOD. I have not been able to find any information on that point. I can simply repeat what Avery and Beans said in their article recently—that they not only applied water to the paris green, but they applied water saturated with carbon dioxid, and they found that the water gradually broke up the paris green, more so in some cases than in others; also that when the water was kept saturated with carbon dioxid inside of a week a large part of the paris green had gone into solution; in other words, the arsenic had been rendered soluble. And they found out that if they ground up the green rather fine, at the end of a week they would have a very large amount of the arsenic going into solution.

Mr. ALWOOD. Let me say that this is the point of my first question. Paris green often scorches the foliage. In a wet season this becomes worse; undoubtedly there is carbon dioxid in the rain water and in the dew, and the solution of the arsenic goes on until the tree may be quite defoliated under some conditions, while under other conditions it will be quite safe. We apply arsenites in large quantities, and it is of vast importance to fruit growers. We use lime constantly, and we mix it with the poison just before we apply it.

Mr. VAN SLYKE. Of course it is known to be true in actual practice that paris green is less injurious when used with lime.

Mr. ALWOOD. We noticed that.

Mr. HAYWOOD. When I spoke just now I did not mean to say that lime did not cause the paris green to be less hurtful. It most certainly does. I simply advanced the proposition, more as a theory than anything else, that paris green is broken up to some extent by lime, and that the calcium salt of arsenious acid or calcium arsenite, although quite soluble in water and greater in amount than the original free arsenic, does not hurt the foliage as much as the small amount of free arsenious acid itself.

The CHAIRMAN (Mr. Wheeler). We will proceed to the consideration

of the next subject in order, the report on dairy products, which will be read by Mr. Van Slyke.

Mr. VAN SLYKE. The report was prepared by the referee, Mr. Le Clerc, in the laboratory at Geneva, and as he has gone abroad since preparing the report, and as Mr. Cavanaugh, the associate referee, who expected to read the report, is absent, I have taken the responsibility of presenting it.

REPORT ON DAIRY PRODUCTS.

By J. A. LE CLERC, *Referee*.

The referee this year divided his work into two divisions:

- I. Determination of albumin in milk.
- II. Detection of renovated butter.

I. DETERMINATION OF ALBUMIN IN MILK

The work on casein in milk has been done so satisfactorily by former referees that the methods of determination of casein as adopted by this association leave nothing to be desired. Not so, however, with the determination of albumin. This determination in the hands of different chemists has never given concordant results. Hence, it was decided to confine our work, in so far as milk was concerned, to the determination of albumin.

Letters asking for cooperation were sent to eighteen chemists. Fourteen replies were received, eleven of which were from men who signified their willingness to cooperate.

On June 21 samples of skim milk, preserved with formalin, were sent to seven men, with the following instructions:

I. (a)^a Remove casein by the official method, Bulletin 46, Revised Edition, p. 55, par. 1.

(b) Albumin, provisional method: Use filtrate of (a), neutralize exactly with potassium hydroxid, and add 0.3 cc 10 per cent acetic acid; heat in water bath till separation of albumin is complete and supernatant liquid is clear. This may require one hour or more. Determine nitrogen in the precipitate.

II. (a)^a Remove casein by precipitation with alum as follows: To 10 grams milk and 50 cc water, at 45° to 50° C., add 1.5 to 2 cc saturated alum solution, filter, and wash.

(b) Albumin in filtrate of (a): Heat in water bath (without neutralization or addition of acid) till precipitate is complete and supernatant liquid is clear. Determine nitrogen in the precipitate.

(c) Albumin in the filtrate of (a): Neutralize filtrate of (a) with potassium hydroxid. Filter off any precipitate of alum hydrate that may be formed. If no precipitation takes place add a few drops of alum solution. Filter precipitate. In either case wash precipitate thoroughly and to the filtrate add 0.3 cc 10 per cent acetic acid. Heat in water bath till supernatant liquid is clear after the precipitation. Determine nitrogen in the precipitate.

III. (a) Penny's method: Place 50 gm milk in 500 cc flask, add 250 to 300 cc water at 45 to 50° C. Add 10 to 12 cc saturated alum solution till casein is completely precipitated. Fill to the mark. Filter.

(b) Heat 100 cc filtrate of (a) as in II (b). Determine nitrogen in albumin precipitate.

(c) Treat 100 cc filtrate of (a) as in II (c). Determine nitrogen in albumin precipitate.

(d) Determine nitrogen in filtrate of (c), which equals nitrogen other than casein and albumin nitrogen.

^a Casein may be determined quantitatively if you have time, in which case determine also the total nitrogen in the milk.

On July 25 another sample of skim milk was sent to four other chemists. The work on albumin by the association began in 1894, when Dr. Van Slyke read a paper on the determination of albumin in milk. The method consisted in heating directly the filtrate of casein (which has been precipitated by acetic acid) about fifteen minutes in water bath. The results always showed that there were other bodies in milk besides casein and albumin, bodies which were neither precipitated by acetic acid nor coagulated on heating the filtrate. These bodies amount in many cases to 10 to 12 per cent of the total protein.

In 1896 the above method was recommended for adoption by the association.

In 1897 the association made a study of the determination of albumin wherein, besides the above method, the acetic-acid filtrate was neutralized (after the precipitation of casein), and then there was added 0.3 cc 10 per cent acetic acid, and the solution then heated on the water bath till albumin was completely precipitated. Results were obtained with the latter modification only from the New York (Geneva) Station, and showed a slightly higher amount of albumin than was obtained by boiling the acetic-acid filtrate direct. It was thought that the large amount of acetic acid might keep some of the albumin in solution, hence, the need of neutralization and the addition of only enough acid to acidify distinctly.

In 1898 the association's work on albumin in milk was changed. Strong solution of tannin was added to the filtrate from casein. Results showed a higher amount of albumin than ordinarily occurs in milk. This is due to the fact that tannin throws down albumoses and peptones, bodies which may be present in milk that is a day old or more.

In 1899 the association continued work with tannin and also tried another method. This was to precipitate the casein by means of alum and then to determine the total nitrogen in the filtrate and call that nitrogen albumin. Obviously, the results were high, because all the noncasein nitrogen can not be assumed to be albumin, unless, perhaps, in fresh milk, and even then the assumption could hardly be absolutely true.

Last year (1900) the association continued the work on the determination of albumin as in the two previous years, but presented one new method. This consisted in precipitating the casein by magnesium sulphate and determining the albumin in the filtrate after the addition of 0.3 cc 10 per cent acetic acid and boiling. This latter method agreed quite closely with the determination of albumin in the filtrate from acetic acid, which was neutralized, 0.3 cc 10 per cent acetic acid, and boiled. By the other methods, where tannin was employed as a precipitant, much lighter results were obtained. In that year it was recommended to adopt the use of tannin as a precipitant for albumin. This recommendation was not adopted, however.

Though the results this year are not so concordant as was hoped for, yet, if the use of tannin as a precipitant for albumin is shown to be erroneous, something will have been gained. This year it was proposed to obtain albumin only by means of heat. I believe, so far as we now know, there is no distinctly albumin precipitant. Heat seems to be the only safe means of precipitating or rather coagulating albumin. Neither albumoses nor peptones nor amid bodies are affected by heat, while both albumoses and peptones are precipitated by tannin.

The proposed methods for the determination of albumin were—

- (1). Neutralize the acetic-acid filtrate (after the removal of the casein by the official method) with potassium hydroxid, adding 0.3 cc 10 per cent acetic acid and heat on the water bath till supernatant liquid is clear.
- (2) Remove casein by use of alum, and coagulate albumin in the filtrate by heat (a) directly, (b) after removal of excess of alum by precipitating it with potassium hydroxid, filtering, and making acid with 0.3 cc acetic acid and heating.
- (3) Repeat (2) with the filtrate obtained by Penny's method.

It has also been the referee's desire to show conclusively that there is a large amount of nitrogen left in the milk after the casein and albumin have been removed, and, to accomplish that, an extra determination was made in the filtrate from albumin.

Following are the results obtained in Sample No. 1.

Determination of albumin in milk, No. 1.

Analyst.	Total protein.	Casein.			Albumin.					Protein other than casein and albumin.
		I (a).	II (a).	III (a).	I (b).	II (b).	II (c).	III (b).	III (c).	III (d).
	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
A. J. Patten, Geneva, N. Y.	3.41	2.69	2.66 2.69 2.68	2.71	0.325 .319	0.240		0.270 .300	No precipitate.	
J. A. Le Clerc, Geneva, N. Y.	3.47	2.79	2.78 2.78	2.66	.262 .275	.287 .280 .319	Slight precipitate.	.287 .312	.312 .312	0.301 .319
C. L. Penny, Newark, Del.	3.42			2.69		.319				.737
A. T. Charron, Ottawa, Canada					.173	.345	.375	.372	.328	.291
C. H. Jones, Burlington, Vt.	3.57	2.79	2.75		.270	.250	.270	.210	.290	.460
		2.79	2.79		.250	.270	.290	.210	.310	.540
		2.78	2.75		.250	.280	.280	.210	.280	
A. L. Knisely, Corvallis, Oreg.	3.41	2.76	2.77		.240	.090	.130	.160	.030	.170
		2.78	2.77		.240	.200	.140	.160	.020	.210
		2.81	2.83		.210	.170	.100	.130	.070	.240
Highest		2.81	2.83	2.71	.328	.345	.375	.312	.312	.540
Lowest		2.68	2.68	2.66	.210	.240	.270	.210	.280	.291
Average		2.76	2.75	2.69	.265	.290	.304	.260	.305	.382

*Omitted from averages.

It will be seen that I (b), II (b), and III (b) give practically identical results, the averages being 0.265, 0.29, and 0.26 per cent albumin, respectively.

The results where the alum was precipitated and filtered are not at all satisfactory. In some cases the alum hydrate appears to retain mechanically nitrogen compounds that are difficult to remove by washing.

The referee made a complete analysis of milk No. 1, using alum to precipitate casein, heating the filtrate directly for albumin, acidifying filtrate with hydrochloric acid and precipitating the albumoses and peptones by means of bromin, and determining the amids in the filtrate from bromin. Ammonia was determined in the milk directly by distillation with magnesium oxid.

Complete analysis of the nitrogenous bodies in milk.

	Per cent.	Per cent of total protein.
Casein	2.78	79.7
Albumin	.28	8.0
Albumose and peptones	.21	6.0
Amids	.21	6.0
Ammonia	.01	.30
Total	3.49	100.00

Work on the proteolysis of milk at the Geneva station shows that whereas the casein constitutes over 80 per cent of the protein in fresh milk 50 per cent of this casein becomes, under certain conditions, converted into albumoses, peptones, and amid bodies.

It would seem necessary, therefore, that our methods for the determination of casein and albumin should give us casein and albumin. It is time that this association should make a more systematic study of the nitrogen compounds of milk. Surely, if we go to a great deal of trouble to determine the albumin, which constitutes from 6 to 8 per cent of the protein, we should give some attention to the determination of the other bodies constituting over 10 per cent of the protein of the milk.

Sample No. 2 was sent out July 25 to six chemists. The only results obtained were from the Geneva station.

This sample of skim milk had been heated, thus reducing below normal the amount of albumin present. However, it answered the purpose of showing that there was almost twice as much noncasein nonalbumin nitrogen as there was albumin, and any method of obtaining the albumin by using tannin as a precipitant, or of calling all the noncasein nitrogen albumin, would give excessively high and erroneous results.

Determination of albumin in milk, No. 2.

Analyst.	Total protein.	Casein.			Albumin.					Protein other than casein and albumin.
		I (a).	II (a).	III (a).	I (b).	II (b).	II (c).	III (b).	III (c).	
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
A. J. Patten, Geneva, N. Y.	2.72	2.28	2.26	2.24	{ 0.135 .151	{ 0.151 .158	{ 0.092	{ 0.158 .151	{ 0.101	0.246
J. A. Le Clerc, Geneva, N. Y.	2.65	2.26	2.28	2.24	{ .115 .125	{ .140	{ .099 .107	{ .158 .158	{ .101 .118	.209 .242
G. E. Patrick, Washington, D. C.*	2.69 2.69	2.19 2.00	{ 2.13 2.06 2.12	{	{ .04 .07	{ .13 .11 .16	{ .07 .09 .07	{	{13 .14	{31
Highest	2.72	2.28	2.28	2.24	.151	.158	.107	.158	.118	.246
Lowest	2.65	2.26	2.26	2.24	.115	.140	.092	.151	.101	.209
Average	2.69	2.27	2.27	2.24	.132	.149	.099	.156	.107	.232

* Received too late to be included in the average.

*Report on official milk work.**

[By T. C. Trescot and D. Stuart.]

	Per cent N.	Per cent, N $\times$ 6.25.	Mean.
Total amount in sample.....	0.43 .43	2.69 2.69	
I. (a) Casein.....	.35 .32	2.19 2.00	2.69
(b) Albumin.....	.006 .011	.04 .07	2.10
II. (a) Casein.....	.34 .33 .339	2.13 2.06 2.12	.06
(b) Albumin.....	.020 .017 .025	.13 .11 .16	2.10
(c) Albumin.....	.011 .014 .011	.07 .09 .07	.13
III. (b) Albumin.....	.025	.16	.08 .16
(c) Albumin.....	.020 .022	.13 .14	
(d) Nitrogen other than casein or albumin.....	.05	.31	.135 .31
Other proteids from I (b).....	.060	.37	
Other proteids from II (b).....	.062	.39	
Other proteids from III (b).....	.062	.39	

* Work done September 25 to October 19, 1901.

II. DETECTION OF RENOVATED BUTTER.

On June 20 the referee sent out samples of renovated and dairy butters, marked simply "A" and "B", to six chemists, with instructions relating to the detection of renovated butter. Only three men sent in results. Most of the methods proposed were those suggested by Messrs. Hess and Doolittle, published in the *Journal American Chemical Society*, March, 1900. The microscopical test was that of Mr. Hummel, to be found in the same journal for June, 1900.

The following are the methods as sent out for comparative trial.

- (1) Try the curd test with both samples A and B. (*Journal American Chemical Society*, March, 1900, p. 151.)
- (2) Appearance of the curd. (*Ibid.*)
- (3) Identification of the source of the curd in A and B. (*Ibid.*)
- (4) Quantitative estimation of the curd (A and B). (*Ibid.*)
- (5) Microscopic test of the curd with polarizing microscope, using selenite plate. (*Journal American Chemical Society*, June, 1900, p. 328; *Bulletin* 46, revised edition, p. 54 (m).)
- (6) Shake 3 to 4 grams A, in test tube, with ether. Pour off fat (dissolved in the ether) into evaporating dish. Do the same with B. Let evaporate spontaneously twenty-four hours or more and compare appearance of residue.

Results of tests for renovated butter.

Test No.	Analyst.	Sample A (renovated butter).	Sample B (dairy butter).
1	A. J. Patten, Geneva, N. Y.	Sputtered	Foamed.
	A. M. Peter and J. O. La Bach, Lexington, Ky.	No foam; acted like oleo.....	Foamed freely, like genuine butter.
	R. W. Thatcher, Pullman, Wash.	No foam; sputtered vigorously.....	Foamed freely.
2	A. J. Patten	Curd separated slowly; melted fat had cloudy appearance.	Melted fat clear.
	A. M. Peter and J. O. La Bach.	Appearance of curd same in both cases; curd granular.	
	R. W. Thatcher	Curd settles slowly and granular, with small lumps.	Melted fat clears quickly; curd smooth and gelatinous.
3	A. J. Patten	Test not satisfactory.	
	A. M. Peter and J. O. La Bach.	Test abandoned.	
	R. W. Thatcher	Filtrate from fat gives flocculent precipitate when boiled with acetic acid.	Filtrate boiled with acetic acid gives slight opalescence.
4	A. J. Patten	Test not satisfactory.	
	A. M. Peter and J. O. La Bach.	Test abandoned.	
	R. W. Thatcher	Per cent casein, 0.54; per cent albumin, 0.062; ratio, 8.78 : 1.	Per cent casein, 0.262; per cent albumin, 0.012; ratio, 21.7 : 1.
5	A. J. Patten	Yellowish-green field mottled with blue, very distinctive.	Uniformly blue field.
	A. M. Peter and J. O. La Bach.	Crystals in both samples, but most characteristic in A.	
	R. W. Thatcher	Blotches of semicrystalline matter visible.	Smooth, clear field.
6	A. J. Patten	Remained clear yellow	Colorless after standing 3 to 4 days.
	R. W. Thatcher	No difference in the curd.	

It will be noticed that the tests are quite characteristic, notably the foam test, and the microscopic test. When samples A and B were sent from Geneva, both samples were in good condition, and could not be distinguished one from the other by any superficial test.

Mr. Patten makes the following comments:

(1) A melted quietly and clear, but on being heated longer, began to sputter, and remained a clear yellow on cooling. B melted with more or less frothing, and became darker in color on cooling.

(2) B on being melted gave a clear supernatant liquid, while with A the curd separated very slowly, giving the melted fat a cloudy appearance. The difference in appearance of the curd was not so distinct, and would not be reliable.

(3) B gave a uniformly colored yellowish-green field. A gave a yellowish-green field mottled with blue, very distinctive.

(4) B became colorless after standing three to four days, while A remained a clear yellow. On dissolving A in ether in the test tube a whitish semifluid substance was left adhering to the sides after pouring off the ether, which substance did not appear when B was treated in the same way.

A. M. Peter and J. O. La Bach say "these results are not very satisfactory, but they appear to show that A is process butter."

R. W. Thatcher comments as follows:

In (3) the temperature at which fat is melted must be kept below 80°C ., or the albumin is coagulated, and does not appear in the filtrate.

(5) Gives a very characteristic distinction, if the butter is kept well cooled before and during the examination.

Recorded results under (4) are those of a single determination, which did not proceed very satisfactorily, hence should not be relied on too greatly.

Hess and Doolittle say that "if the artificial curd of process butter has been derived from milk, then the ratio of the percentage of casein to the percentage of albumin should be the same as that ratio is in milk, or about 9 parts casein to 1 part of albumin." Mr. Thatcher actually found the ratio to be 8.78:1, but, as he says, this is not to be relied upon too greatly.

It was the referee's intention, when the results were all in and comments noted, again to send out samples, this time using only those methods which appeared to give characteristic results, and also to give trial to any suggestions which might be offered by the coworkers.

Farmers' Bulletin 131, by G. E. Patrick, confirms the foam test, which has been successfully used for the past ten years for the detection of oleo.

The above results will only show that the sample under examination is or is not genuine butter; but if it is not genuine butter, the ordinary chemical methods for the examination of the fat will at once show whether the sample be oleo or renovated butter.

RECOMMENDATIONS.

First. Continued work on determination of albumin in milk is suggested.

Second. Continued work on adulterations of butter is advised.

President Van Slyke resumed the chair.

The PRESIDENT. Mr. Patrick began some work on the subject of renovated butters some time before it was taken up by the referee, and his results cover the ground more thoroughly, probably, than the report of the referee, and he will, in accordance with my request, give his paper separately.

Mr. PATRICK. But as a part of the referee's report. It is supposed to be handed in to him, but it was too late in finishing.

Mr. STREET. I have a resolution that I would like to offer, if in order, and ask that it be referred to the committee on resolutions for favorable report:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

Mr. LEACH. Mr. President, may I say a word with regard to the systematic examination of preservatives for milk? It seems to me that the subject is one that we ought to consider carefully, in view of the marked increase of late in this form of adulteration. In the Massachusetts Board of Health laboratory we examine milk collected from all parts of the State, with the exception of Boston, which has a very efficient system of its own. During the summer months, in addition to our regular analysis of milk for solids and fat, we examine for pre-

servatives every sample of milk that is brought in, amounting to some 500 samples per month, and varying from 20 to 60 per day. This is not as difficult as at first sight appears, if reduced to such a system as I have adopted.

I might say at the outset that in 1900, for instance, from June to September, inclusive, upward of 2,000 samples were examined, of which about 3 per cent contained formaldehyde, approximately one-half of 1 per cent contained boracic acid, and a very few samples contained carbonate of soda. My method of procedure is as follows:

The milk samples are arranged in the order of their numbers, and about 10 cc of each is poured into a casserole, the casseroles being arranged in the same order as the containers of the samples. Each casserole is then treated alike in succession, first adding from a large stock bottle fitted with a siphon about 10 cc of concentrated hydrochloric acid containing a very little ferric chlorid, then holding the casserole over the gas flame and giving it a rotary motion while heating. The well-known violet coloration, indicative of formaldehyde, will appear if as much as 1 part in 300,000 is present. Less than one minute is required in making this test on each sample.

I am aware that this method has been the subject of some criticism, due doubtless to the fact that various aldehydes when present in milk give color reactions under this treatment. I have, however, found nothing but formaldehyde that would give the peculiar violet color, which is very distinctive. I have tested its reliability again and again during the last five years, and while using it only as a preliminary test, I may say that I have always been able to confirm it.

The tests for boracic acid and carbonates are made incidental, as it were, to the cleaning of the platinum dishes in which the determinations of total solids were made, so that very little extra time is involved. The residues are first incinerated in the original numbered dishes, which are afterwards arranged in order on a tray. With a pipette, one or two drops of dilute hydrochloric acid are added to each ash in succession, noting effervescence, which, of course, indicates carbonate, to be confirmed later by the rosolic acid test.

By means of a wash bottle a few centimeters of water are added to each acidulated milk ash, the tray is given a rotary motion to aid in dissolving the ash, and a strip of turmeric paper is first immersed in each solution and then allowed to dry while adhering to the side of the dish, out of contact with the liquid. The usual cherry-red color on the paper when dry indicates boracic acid.

Thus the three preservatives most commonly used are very readily singled out, confirmatory tests to be afterwards made, of course, on fresh samples of the milk.

THE PRESIDENT. I do not know but that Mr. Patrick's paper had better be put over until after recess, when there will be no danger of interruption. If there is no objection, we will have this paper the first thing this afternoon.

MR. PATRICK. Before we leave the subject of analysis of milk I would like to suggest that perhaps the different results, of which the president spoke, obtained by the chemists, in the determination of albumin and casein, might even in the case of those preserved samples have been due to enzymes—to changes produced by the enzymes. I have not seen the figures, but the reason I suspected this was, that in

our laboratory, in the work being done by Mr. Stuart and Mr. Trescott, the figure for albumin became exceedingly low, about 0.13 per cent. I presume that that is the low figure, or one of them, that the referee referred to. Those analyses were not made until a few weeks ago—the latter part of the summer or the early fall. The samples had remained in a refrigerator, but a great many degrees above freezing, and it occurred to me that the low albumin might have been caused by the action of some enzym, for we know that whereas some of these preservatives will prevent the action of bacteria and germs, they do not in anywhere near the same degree prevent the action of the enzymes. I merely suggest this as a possible explanation.

Mr. Wheeler then presented the report of the committee on amendment of the constitution, as follows:

REPORT OF COMMITTEE ON AMENDMENT OF THE CONSTITUTION.

The committee reports favorably upon the amendment proposed to section 4. That section now reads:

There shall be appointed by the president at the regular annual meeting a referee and associate referee for each of the subjects to be considered by the association.

As amended the section will read:

There shall be appointed by the executive committee at the regular annual meeting a referee and associate referee for each of the subjects to be considered by the association.

Mr. Ewell moved to amend this section further by changing that portion after the word "referee" so as to make it read, "and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate."

The latter amendment was accepted, and the original amendment, thus changed, was adopted, making the section read as follows:

There shall be appointed by the executive committee at the regular annual meeting a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

The committee reports favorably upon the amendment proposed to section 7, which section reads:

No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

I would say that this section was drawn up at a time when we devoted ourselves almost exclusively to fertilizer analyses; but since that time we have added analyses of cattle foods and analyses of human foods, and the amendment was proposed in order to embrace those two subjects as well as the subject of commercial fertilizers. The section will read after amendment:

No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

The amendment was adopted.

The committee also reports favorably upon the amendment proposed to section 2, to strike out the part reading—

Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution, and to insert in place of it the following:

All persons eligible to membership shall become members *ex officiis* and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials.

The PRESIDENT. I suppose that if the constitution had been strictly interpreted not a single one of us would really be a member. I never signed the constitution in my life, and I do not know of anyone else who has.

Mr. EWELL. That is an amendment that will save us considerable trouble in the Department of Agriculture, because we get so many inquiries. In our replies we have always interpreted the constitution as though the words of this amendment were actually a part of it.

The amendment was adopted.

Mr. Wheeler then presented the report of the committee on resolutions.

REPORT OF COMMITTEE ON RESOLUTIONS.

The adoption of the following resolution which was offered yesterday is recommended:

Resolved, That the Association of Official Agricultural Chemists welcomes the action of the National Association of State Dairy and Food Commissioners in appointing a committee of its members to represent to our association its views upon the subject of food standards—the formulation of which has already been undertaken by this association for the guidance of its members—and upon the methods of detection of food adulteration.

Adopted.

The following resolution was presented this morning:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

The committee recommends the following substitute:

Resolved, That it is the sense of this association that, until a proposed new method or modification of an existing method has been tested by the referee with favorable results, it shall not be submitted by him for general test by the association.

It is thought that this form would be as mandatory, that the referee would be expected to do what the association had expressed as its sense in the matter, without urging it upon him.

The substitute was adopted.

At 12.30 the convention adjourned until 2 p. m.

FRIDAY—AFTERNOON SESSION.

President Van Slyke called the meeting to order at 2 o'clock.

The PRESIDENT. We will continue the paper that was about to be presented when we adjourned—Mr. Patrick's paper on butter.

Mr. PATRICK. In the paper that I have to present as part of the report of the referee I have introduced a number of results from samples that were not sent out by the referee—samples that were used in the course of our somewhat extended study of renovated butters in the Bureau of Chemistry.

REPORT ON THE IDENTIFICATION OF RENOVATED BUTTER.

By G. E. PATRICK.

I. THE BOILING TEST.

This test I have made usually with aid of a test tube instead of a spoon, sometimes resorting to the latter, however, for corroboration or for a more distinct result as to "sputtering" in doubtful cases. I put into a 6-inch tube enough of the butter to make a depth of $1\frac{1}{4}$ to $1\frac{1}{2}$ inches when melted, and boil it intermittently over a free flame, boiling it briskly for six or eight seconds, then removing it from the flame for an equal or greater length of time—long enough to be certain of its behavior—and repeating the operation until all the water is expelled. If the butter be genuine the time arrives when, after removal from the flame, it boils up in small bubbles, producing a foam which rises to a greater or less height, sometimes even overflowing the tube. Renovated butter produces much less foam, if any (oleomargarine none) and of course boils more noisily than does genuine butter.

From late experience I am inclined to believe that in doubtful cases the spoon form of the test is more reliable than the test-tube form.

This test—i. e., the boiling test in some form—being well known and in general use, I will report results only on the referee's samples A and B, and four others that accompany them throughout all the tests.

TABLE I.—*Results of boiling test.*

Sample A (No. 22608); boiled noisily, producing but little foam.
 Sample 23142, process; boiled noisily, producing but little foam.
 Sample 23144, process; boiled noisily, producing but little foam.
 Sample B (No. 22609); boiled more quietly, producing abundance of foam.
 Sample 23143, creamery; boiled more quietly, producing abundance of foam.
 Sample 23145, creamery; boiled more quietly, producing abundance of foam.

II. THE HESS AND DOOLITTLE METHOD—APPEARANCE OF THE CURD AFTER DRYING ON A GLASS PLATE (MICROSCOPIC SLIDE).^a

My first trials of this test (April, 1901) were an entire failure, because, having simply followed the authors' directions and poured the fat-free curd and brine in a thin layer upon the glass plates, the salt of the dried mass practically hid everything else from view.

In subsequent trials more satisfactory mounts were obtained by judiciously diluting the curd before spreading upon the slides; but since the brines differ greatly in their

^aJour. Am. Chem. Soc., Mar., 1900, p. 150.

content of salt, it is impossible to know before trying how much dilution is necessary in each case. Very great dilution is evidently to be avoided, as the casein particles that lodge upon the slide are then too few in number.

This mode of procedure, viz, diluting the curd according to judgment, always with at least several times its volume of water, before pouring upon the slides, was followed in all of the tests here recorded and in nearly all that we have made. But even with considerable experience in preparing the mounts, we frequently find them hardly satisfactory after drying, either from too much salt or because the layer of curd is too thin.

In one series of over forty samples, where we had made the layers too thick, I tried to improve them by dissolving off the salt by gentle immersion in water for a minute or so, but the result was not a success; too much besides the salt was removed or else the curd was left too transparent.

In another set, however, of four genuine and four process butters, another and more successful variation of the method was tried, viz, that of diluting the fat-free curd with considerable water, allowing to settle, and then spreading upon the slide a small portion drawn from the bottom by means of a glass tube or inverted pipette. The results on these few samples were so satisfactory that we shall try this mode again at the first opportunity. Theoretically, we should be able to escape all trouble from salt crystals in this way.

Working by the present method, however, one has often much trouble in interpreting the appearances presented by the curd. The majority of process butters, in our experience, give quite distinctly the appearance described by Hess and Doolittle, viz, a coarse or distinctly granular curd; but some samples certainly do not present this appearance distinctly. And, on the other hand, we have met with a few low-grade butters, "country butters" and "farmers' rolls," undoubtedly genuine, showing casein granules of such size and number, intermingled with fine matter of course, that if judged by this method alone they would be strongly suspected of being process butter, at least in part. In fact some samples of this class have appeared more like process butter than some few other samples known to be process butters, as shown in the following table:

TABLE II.—*Identification of process butters by the Hess and Doolittle method.*

Laboratory No.	Interpretation of appearances with—		Character of butter, as known from sources, verified by other tests.
	Power 85 diameters.	Power 7 diameters.	
22537	Suspicion of process	Suspicion of process	Country butter; packing stock.
22538	Genuine	Genuine	Do.
22539	do	Probably genuine	Do.
22540	do	Genuine	Do.
22541	do	do	Do.
22542	do	do	Do.
22549	do	do	Do.
22550	do	do	Do.
22551	do	do	Do.
22552	do	do	Do.
22553	Suspicion of process	Probably genuine	Do.
22554	do	Suspicion of process	Do.
22610	Probably genuine	Genuine	Do.
22342	Process	Suspicion of process	"Farmer's Roll," low grade.
22343	Genuine	Genuine	Do.
22345	Suspicion of process	do	Do.
22346	Probably genuine	do	Do.
22545	do	do	"Dairy Print," good quality.
22546	Genuine	do	Do

TABLE II.—*Identification of process butters by the Hess and Doolittle method—Continued.*

Laboratory No.	Interpretation of appearances with—		Character of butter, as known from sources, verified by other tests.
	Power 85 diameters.	Power 7 diameters.	
22547	Suspicion of process.....	Genuine.....	"Dairy Print," good quality.
22563	Process.....	do.....	Do.
22349	Suspicion of process.....	do.....	Do.
22543	Genuine.....	do.....	"Dairy Tub," good quality.
22544	do.....	do.....	Do.
22559	do.....	do.....	Creamery, good quality.
22564	do.....	do.....	Do.
22561	Suspicion of process.....	Probably genuine.....	Do.
22210	Genuine.....	Genuine.....	Ladled.
22214	Process.....	Suspicion of process.....	"Ladled;" appears to be in part process. ^a
22217	Suspicion of process.....	Genuine.....	Ladled.
22218	Genuine.....	do.....	Do.
22333	do.....	do.....	Do.
22335	do.....	do.....	Do.
22336	do.....	do.....	Do.
Ottumwa.	do.....	do.....	Do.
22555	Process.....	Suspicion of process.....	"Process" or renovated.
22558	do.....	Probably process.....	Do.
22560	Suspicion of process.....	Probably genuine.....	Do.
22208	do.....	Suspicion of process.....	Do.
22209	do.....	Genuine.....	Do.
21938	Process.....	Process.....	Do.
21816	Probably process.....	do.....	Do.
21817	Process.....	do.....	Do.
21794	do.....	do.....	Do.
21937	do.....	do.....	Do.
21944	do.....	do.....	Do.
22562	do.....	do.....	Do.
A (22608)	do.....	do.....	Do.
B (22609)	Probably genuine.....	Genuine.....	"Dairy."
23142	Process.....	Probably process.....	Process or renovated.
23144	Suspicion of process.....	Probably genuine.....	Do.
23143	Genuine.....	Genuine.....	Creamery.
23145	do.....	do.....	Do.

^a Microscopic appearance strongly indicates admixture of "process."

Three different magnifying powers were used in examining the slides—a reading glass of low power, a microscopic combination enlarging 7 diameters (Hess and Doolittle employed 3 to 6 diameters), and another enlarging 85 diameters. The first was quite insufficient; the second and third each had their advantages, but a comparison of the known character of the butters with the conclusions drawn from examination with the two powers independently, and usually without knowledge of the character of the sample, shows the lower power to be in general the more suitable of the two. The higher power (85 diameters) sometimes misled by giving an exaggerated idea of the size of minute particles of casein, while on the other hand with a few samples of very smooth "process" it gave the more reliable indications. But, as the table shows, with the use of either power there was often a considerable degree of uncertainty in the conclusions, which I can hardly believe due to lack of care in making the observations or in preparing the slides. With the lower power (7 diameters) the conclusions are seen to have been positively or "probably" wrong only three times

out of the fifty-three (each time on process butter); "suspicious" of what was not the fact three times; and "suspicious" (instead of being positive) of what was the fact three times. In twelve instances the appearances were so indecisive that only qualified verdicts could be rendered, and it is altogether probable that in some of these, at least, another observer would have reached other conclusions. To test this point (the personal equation of different observers) Mr. Van Dyke and Mr. Stuart made independent observations, with magnifying power of 85 diameters, on 40 samples (2 slides each), with the result that their conclusions, although expressed in some 12 cases as probabilities or suspicions, were to all intents the same in thirty-one out of the forty; somewhat different in four and exactly opposite in five instances. Their results would probably have been more concordant had the lower power of microscope been used; but the fact that different observers are likely to sometimes interpret differently the appearances upon the same slides is illustrated.

From all of our experience I conclude that while there is room for much improvement in this test, even as it stands, it is to be classed among the useful corroboratory tests available to the chemist.

III.—THE HESS AND DOOLITTLE METHOD FOR THE IDENTIFICATION OF THE SOURCE OF THE CURD BY QUALITATIVE TEST FOR ALBUMINS IN SOLUTION.

We have usually begun by at once dissolving the fat in ether without the preliminary melting and decanting of the bulk of the fat, as described by Hess and Doolittle, a variation that can not possibly have affected the tests upon the washed curd and brine, and which was adopted in order to bring the test into line with the *quantitative* curd test, and also to observe under identical conditions the behavior of the curds in regard to settling from the ether, or, more accurately, to roughly compare the amounts of that part of the curd which has the property of remaining long suspended in ether. Hess and Doolittle state that the curd of genuine butter remains suspended in ether for a considerable time.

In our experiments we have not attempted to wait for a perfect subsidence, but in each set of experiments have allowed to all the samples the same time (never more than one and one-half hours, and usually only 5 to 15 minutes) before decanting off the more or less turbid ether and adding a new portion. Proceeding in this way we found that, as a rule, at the second, third, or fourth treatment with ether, depending upon the amounts used, the remaining curds of process butters would settle immediately after stirring, leaving the ether perfectly clear, while with genuine butters treated in exactly the same way the ether would be cloudy and remain so for a long time. The treatment with ether being repeated until the sixth time, in one set of experiments, the curds of some of the genuine butters showed signs of being depleted of this particular form of matter, the ether becoming quickly clear or nearly so.

But it must be stated that while in our experience process butters nearly always behave in the manner described, we have seen several samples of genuine butter behave in exactly the same way. Therefore, while these observations are of no immediate value they may point out a line for future study of a more critical character.^a

^a Further experiments made since the above was written show the phenomenon in question to be dependent merely, or at least mainly, upon the relative dryness of the curds. As might be expected, a wet curd yields less matter suspensible in ether than does the same curd when dry; and the behavior of any butter, genuine or renovated, can be entirely changed, either by air, drying it for several days in a thin layer, or by intimately mixing 10 to 15 per cent of water with the softened or melted butter.

Coming now to the test of the fat-free curd and brine, these having been diluted with a little water and the soluble portion filtered off, the clear filtrate was treated as Hess and Doolittle direct, viz, by acidifying slightly and boiling. It soon became evident, however, that the greater part of the precipitate obtained from process butters was thrown down by simple acidification, without heat. Therefore, for the sake of investigation, it has been my habit to first remove this precipitate, if any were found, and then to test the filtrate for albumin by heat. In this way I have found the albumin to be only a very minute quantity in either genuine or process butter, not nearly enough to serve as the basis for a distinguishing test. In fact, in a few samples of very rancid process butter albumin has been practically absent.

From all this it seems quite evident that the real basis of the Hess and Doolittle test is the soluble (uncoagulated) casein rather than the albumin of the brine; this, however, is nothing against the test.

Passing on to the results obtained with it, without attempting to give in detail all of the individual trials, we can say from our experience, which has been mainly with stale and rancid samples, that process butters of that description are very likely to be passed as genuine by this test, their soluble casein being often no greater in amount than that of genuine butters, and sometimes being practically absent. This may be illustrated with a set of four process and four genuine butters tested last April. The former had been kept in the refrigerator nine to twelve weeks after purchasing. Three of the latter were considerably older.

In these trials the soluble casein and the albumin were precipitated together exactly as directed by Hess and Doolittle. The amounts taken were 100 grams each.

TABLE III.—*Qualitative test of brines; soluble casein and albumin together.*

Genuine:

- 21251. Distinct milky precipitate; slow to settle.
- 21288. Mere trace; practically nil.
- 21289. Small distinct precipitate which settles.
- 21800. Very slight precipitate; less than in 21289.

Process:

- 21794. Very slight precipitate; less than in 21251 or 21289.
- 21795. Quite a precipitate; probably twenty times that from the genuine butter.
- 21802. Heavy precipitate; perhaps twice that of 21795.
- 21803. Merest trace; the least of any except 21288.

Two of the process butters would have been so declared by the test and two would not.

In Table IV is given the result with much older samples, showing the practical absence of both soluble casein and albumin in certain old process butters, and illustrating another fact not previously mentioned, viz, that some genuine (?) butters contain enough soluble casein to cause them to be either condemned by the test or regarded as very suspicious. All such samples that we have found are included in the table. With one exception they are "country butter," the "packing stock" of ladlers or renovators.

Theoretically they were probably churned from cream not very much ripened, were certainly not well freed from buttermilk at churning and were salted sufficiently to prevent, largely, the coagulation of the casein with the lapse of time. That they are exceptional among genuine butters is evident from the fact that they are the only ones giving so strong a test for soluble casein in a list of 40 genuine butters tested at one time. The other 33 (not included in the table) showed only sufficient soluble casein to be described by the words "opalescence," "faintest opalescence," "faint turbidity," and in a few cases—six—"small or light precipi-

tate." The albumin precipitates differed somewhat among themselves, but all were very small. The samples had been in refrigerator nearly all the time since their purchase; the length of such storage is stated in the table.

TABLE IV.—*Tests for soluble casein and albumin with old butters (Hess and Doolittle method).*

No.	Character.	Age since purchase.		Test for soluble casein.	Test for (soluble) albumin.
		Mos.	Days.		
21794	Process	9	7	Considerable precipitate settled.	Slight precipitate (or cloudiness).
21816	do	8	4	Nothing	Do.
21817	do	8	4	Heavy precipitate.....	Do.
21938	do	8	3	Light precipitate settled	Do.
22208	do	6	8	Very light precipitate settled.	Do.
22209	do	6	7	Faint opalescence.....	Do.
22555	do	4	9	Nothing	Slight precipitate settled.
22556	do	4	9	do	Nothing. ^a
22558	do	4	9	do	Do.
22560	do	4	9	do	Do.
22538	Country butter ^b ..	4	12	Heavy flocculent precipitate.	Slight precipitate (or cloudiness).
22539	Country butter...	4	12	Very heavy flocculent precipitate.	Do.
22540	do	4	12	do	Do.
22549	do	4	9	Heavy precipitate.....	Slight precipitate.
22551	do	4	9	Considerable precipitate settled.	Do.
22554	do	4	9	Heavy precipitate.....	Do.
22559	Creamery.....	4	9	Considerable precipitate settled.	Do.

^a The tests recorded in this table were upon only 16 to 17 grams of butter. In larger amounts perhaps albumin might have been found. Casein was precipitated by 0.3 to 0.5 cc. of 10 per cent acetic acid; the filtrate from casein was merely heated for albumin.

^b Or packing stock.

Results upon the referee's samples A and B, and the four that were tested with them throughout, were as follows:

TABLE V.—*Tests on samples of referee and comparative samples for soluble casein and albumin.*

FIRST TEST, SEPTEMBER 25.

Sample. ^a	Test for soluble casein.	Test for albumin.
A (22608). Process	Nothing	Very slight precipitate.
B (22609). Dairy	Mere trace.....	Opalescence.
23142. Process.....	Heavy precipitate....	Very slight precipitate.
23144. Process.....	do	Opalescence.
23143. Creamery	Trace.....	Very slight precipitate.
23145. Creamery	Opalescence	Do.

^a Samples A and B were received July 5, and had been held in refrigerator. The other four were entirely fresh, purchased September 24.

TABLE V.—*Tests on samples of referee and comparative samples for soluble casein and albumin—Continued.*

SECOND TEST, NOVEMBER 7.

Samples.	Test for soluble casein.	Test for albumin.
A (22608). Process	Nothing	Very small precipitates or turbidities for all.
B (22609). Dairy	Opalescence	
23142. Process	Small precipitate	
23144. Process	Opalescence	
23143. Creamery	...do	
23145. Creamery	...do	

Here it is seen that in the six weeks elapsing between the first and second tests practically all the soluble casein of 23144 and a large part of that of 23142 had disappeared, having become coagulated, no doubt.

In the process butter A this coagulation had entirely taken place before the date of the first test. Samples A and B were in refrigerator during the six weeks between the tests, the other four were in a dark closet in the laboratory. Both tests were of course made on the same quantities of butter—in this case 25 grams.

It should be stated here that a process butter made in the laboratory by incorporating clear butter fat with buttermilk two days from the churn gave a negative test for soluble casein, as would be expected. Therefore, if sour buttermilk or curdled milk of any kind be used to the exclusion of sweet milk in the making of process butter, even the fresh article would not be detected by this test.

I am informed that buttermilk is used by some of the "process" manufacturers, but whether it ever wholly replaces sweet milk I have not learned, nor am I informed as to the extent to which the "ripening" of the milk is carried before its incorporation with the butter fat; this would of course affect the results.^a

Oleomargarine, it is known, is sometimes made with buttermilk alone, and a fresh sample of such oleomargarine submitted to this test gave the ideal result for genuine butter. The test would of course have been the same had it been a process butter made in a similar way.

IV. QUANTITATIVE ANALYSIS OF THE CURD AND BRINE (HESS AND DOLITTLE METHOD).

After what has been said concerning the qualitative testing of the curd and brine, it is only necessary here to give the results of the quantitative analyses made.

All the determinations of nitrogen in the various precipitates and residues were made by Mr. T. C. Trescot, of the Bureau of Chemistry.

In our first set of trials the soluble casein and albumin were thrown down together, exactly as directed by Hess and Doolittle. The use of the separatory funnel, as directed by them, was found not only needless but objectionable, because of adhesion of casein thereto, and was not used in subsequent trials. Fifty grams of^b each butter were taken.

^a More extended experience, since the above was written, with the process butters as found in the Washington market, shows that a large proportion of these butters, even while strictly fresh, have their casein largely or wholly in the coagulated state (insoluble casein); and since most of them are doubtless made with skimmed or whole milk, and not with buttermilk, the writer infers that the ripening of the milk (for flavor) is often carried so far that the casein curdles very soon thereafter—quite likely when the milk is mixed with the hot or warm butter-fat.

^b In later work only 25 grams were used. Even with this amount the filtration of curd is often very slow.

Results were as follows, together with the ratios regarded by Hess and Doolittle as the ratios of "casein to albumin:"

TABLE VI.—*Determination of nitrogen (Hess and Doolittle method).*

Sample.	Nitrogen of insoluble casein.	Nitrogen of soluble casein and albumin.	Ratio of insoluble casein to soluble casein and albumin.	Age of butter from time of purchase.
	Per cent.	Per cent.		Mo. dys.
21796. Process	0.102	0.009	11.4 : 1	2 25
21797. Process	.102	.003	36.5 : 1	2 25
21804. Process	.098	.006	15.8 : 1	2 11
21806. Process	.141	.002	69.9 : 1	2 8
21816. Process	.046	.006	7.5 : 1	1 22
21805. Oleomargarine	.082	.003	29.3 : 1	2 8
21807. Creamery	.049	.001	43.7 : 1	2 8

The ratio of 8.6 : 1 mentioned by Hess and Doolittle is approached by only two of the process butters, while two others furnished ratios averaging higher than that of the known creamery butter.

Later, in examining another set of samples, we separated the soluble casein from the albumin, precipitating the former by acetic acid in the cold, and the latter, after neutralizing the liquid, with 0.3 cc of 10 per cent acetic acid and heat.

In the filtrates from albumen the proteids and other matters precipitable by tannin were thrown down by that agent in the usual manner, and the amounts were considerable, as shown in Table VII.

TABLE VII.—*Determination of nitrogen (Hess and Doolittle method).*

Sample.	Percentage of nitrogen in form of—				Ratio of insoluble casein to soluble casein and albumin.	Ratio of insoluble casein to soluble casein, albumin, and tannin precipitate (protein of).	Ratio of total casein to albumin and tannin precipitate (protein of).
	Insoluble casein.	Soluble casein (by acetic acid, cold).	Albumin (by acetic acid and heat).	Tannic acid precipitate.			
22555. Process	0.011	0.039	Mere trace	0.037	0.28 : 1	0.14 : 1	1.35 : 1
22556. Process	.012	.012	do....	.004	1.0 : 1	.75 : 1	6.0 : 1
22558. Process	.022	.037	None	.029	.60 : 1	.33 : 1	2.0 : 1
22560. Process	.021	.010	Mere trace	.005	2.1 : 1	1.5 : 1	6.2 : 1
22548. Oleomargarine	.031	.038	None	.010	.82 : 1	.65 : 1	6.9 : 1
22566. Oleomargarine	.095	.008	Mere trace	.005	11.9 : 1	7.3 : 1	20.6 : 1
22544. Dairy tub	.066	.000	0.008	.008	8.25 : 1	4.0 : 1	4.0 : 1
22545. Dairy print	.025	.049	Mere trace	.025	.51 : 1	.33 : 1	3.0 : 1
22546. Dairy print	.031	.046	do....	.034	.67 : 1	.39 : 1	2.3 : 1
22563. Dairy print	.031	.017	do....	.024	1.8 : 1	.75 : 1	2.0 : 1
22559. Creamery	.021	.029	do....	.012	.72 : 1	.51 : 1	4.2 : 1
22564. Creamery	.008	.011	0.013	.024	.33 : 1	.17 : 1	.51 : 1

All of these samples had been purchased approximately two and one-half months prior to the trial, and had meantime been stored in the refrigerator.

In the first column of ratios are those called for by the Hess and Doolittle method, and which according to these investigators should be about 8.6 : 1 for process and much greater for genuine butters. A study of the table shows that neither this nor any other ratio can be found, in either of the three columns of ratios, distinguishing the one class of butters from the other.

Next we tried the method upon the absolutely fresh butters referred to above under the qualitative test, two genuine and two process, together with the referee's samples A and B, which had been held in the refrigerator since their arrival two months and twenty-one days before the trial was made. Duplicate analyses of the curd were made, the results being shown in Table VIII.

TABLE VIII.—*Determination of nitrogen in samples of referee and comparative samples (Hess and Doolittle method).*

Sample.	Percentage of nitrogen in form of—				Ratio of insoluble casein to soluble casein and albumin.	Ratio of insoluble casein to soluble casein, albumin, and tannin ppt. (proteid of.)	Ratio of total casein to albumin and tannin ppt. (proteid of.)
	Insoluble casein.	Soluble casein (by alum).	Albumin (by acetic acid and heat).	Tannic-acid precipitate.			
A (22608). Process.....	{(1).... 0.073	0.005		0.008	14.6 : 1	5.6 : 1	
	{(2).... .075	0.011	0.000	.004	6.8 : 1	5.0 : 1	21.5 : 1
B (22609). Dairy	{(1).... .033	.010		.010	3.3 : 1	1.6 : 1	
	{(2).... .031	.018	.000	.006	1.6 : 1	1.3 : 1	8.1 : 1
23142. Process	{(1).... .009	.050		Lost. ^a	.18 : 1	.16 : 1	
	{(2).... .008	.055	.002	Lost. ^a	.10 : 1	.12 : 1	6.3 : 1?
23144. Process	{(1).... .010	.065		.010	.15 : 1	.13 : 1	
	{(2).... .009	.076	.002	.006	.12 : 1	.11 : 1	10.6 : 1
23143. Genuine	{(1).... .018	.021		.011	.86 : 1	.56 : 1	
	{(2).... .020	.029	.007	.006	.56 : 1	.48 : 1	3.8 : 1
23145. Genuine.....	{(1).... .040	.011		.009	3.6 : 1	2.0 : 1	
	{(2).... .040	.021	.003	.006	1.7 : 1	1.3 : 1	6.8 : 1

^a Assumed as 0.008 in calculating ratios.

Here again we look in vain for any uniformity of ratios by which to distinguish the two classes of butters.

Neither is there seen any marked difference between the fresh and the stale genuine butters; but the fresh process butters show percentages and ratios quite different from those of the stale "process" of this and the preceding tables, because of the much greater proportion of soluble casein; i. e., the still uncoagulated casein of the milk used in their manufacture.

Thus the conclusions drawn from our experience with the qualitative test and stated above are fully borne out by the figures obtained in the quantitative work.

V. MICROSCOPIC EXAMINATION OF BUTTER WITH AND WITHOUT POLARIZED LIGHT AND SELENITE PLATE.^a

While some process butters have been found showing imperfect crystallization, giving the appearance of white flakes, or in lower degree of frostiness or haziness when viewed by polarized light with crossed nicols, quite a number of samples have been encountered during the past year that were nearly or quite free from these appearances, quite as much so as rather old samples of genuine butter frequently are, especially if they have been misused in regard to temperature.

In fact, a very considerable number of process butters have been found much freer from these appearances than were certain samples of genuine butter (canned) that had traveled in the Tropics, and whose frosty and flaky appearance at once raised suspicion of adulteration with foreign fats or, at least, of "processing," both of which suspicions were fully removed by a critical study of the butters. The fact seems to be that in the present practice of the best processing factories the cooling of the melted fat is effected so quickly that even incipient crystallization is almost entirely prevented; in fact, practically speaking, is prevented. When less care is taken in this step of the process the product is, of course, more easily detected by the method in question. Fresh samples are doubtless more free from these appearances than are old ones. As to colors with the selenite plate, the purple mottling which at one time I supposed to be a sign of "process" I have ceased to regard as of any significance, since it may occur in almost any sample of butter where rancidity is developing; but, strange to say, it is once in a while nearly absent in very rancid samples.

An important feature of the microscopic examination of butter is the observation of the casein, which occurs either nearly dry or inclosed in what appear to be minute pools or droplets of brine.

In process butters especially these casein particles are usually much larger, as well as more numerous, than in most genuine butters; but I have found them of large size and in considerable abundance in a number of "country butters," some of which are mentioned in connection with the microscopic examination of curd in the Hess and Doolittle test.

Probably any butter from which the milk is not well washed at churning will show these large casein spots. In my experience good creamery butters are very nearly free from them. These spots are almost always of a pale yellow color, sometimes of a dirty white (rarely white or colorless) when viewed with a full illumination, becoming white and gelatinous in appearance when viewed without sub-stage illumination; that is, upon simply turning away the mirror.

In fact, when viewed in this way many small casein spots frequently become visible that were not noticeable with full illumination. This examination is best made with ordinary light, the light thus being better and the field larger than when the nicols are attached.

Magnifying powers of 85 or 120 diameters give good results. With process butters the pale yellow casein spots are usually the most striking and the most characteristic objects in the field, but several mounts should be examined, as the casein is often not evenly distributed. Considering the importance of this observation of the casein, I venture to include it in my report, although not called for by the referee.

^a Journal Am. Chem. Soc., June, 1900, p. 328.

TABLE IX.—*Microscopic test of butters with and without polarized light.*

[Magnification, 85 diameters.]

Sample.	With substage illumination.	No substage illumination.	Polarized light; crossed nicols.	Polarized light, with selenite plate.	Conclusions and remarks.
A. Process....	Many small and some large casein spots of dirty yellow color. B. fat rosettes.	Much casein; gelatinous.	Haziness, frostiness, flakes, imperfect and perfect fat crystals.	Fat crystals, variegated colors. Purple mottles on blue field.	The abundance and size of casein spots indicate renovated or low-grade dairy butter. The fat crystals suggest renovated butter.
B. Dairy	A few pale yellow casein spots, mostly small; normal.	A few casein spots, gelatinous; normal.	No crystals, no flakes, no frostiness; normal.	Nearly solid colors. Little purple mottling on blue field.	The casein spots might come from admixture of renovated butter; they call for further study of sample.
23142. Process.	Many large casein spots, pale yellow.	Many large casein spots, gelatinous.	No crystals, no flakes, no frostiness; normal.	Solid colors, no crystals, no mottling; normal.	The numerous and large casein spots indicate renovated or low-grade dairy butter.
23143. Creamery.	Normal.....	Very few and very small casein spots; normal.	No crystals, no flakes, no frostiness; normal.	Faint purple spots on blue field; normal.	Genuine butter.
23144. Process.	Many large casein spots, pale yellow.	Many large casein spots, gelatinous.	No crystals, no flakes, no frostiness; normal.	Slight purple mottling on blue field; normal.	The numerous and large casein spots indicate renovated or low-grade dairy butter.
23145. Creamery.	Normal.....	Practically no casein spots; normal.	No crystals, no flakes, no frostiness; normal.	Faint purple spots on blue field; normal.	Genuine butter.

VI. THE "CREEPING TEST."

The "creeping test," as I venture to call it for brevity, the writer had never tried nor heard of prior to receiving the referee's circular, and therefore has little experience to offer. Four grams of the butter were dissolved in 20 cc ether and filtered into 3-inch porcelain evaporators. The appearance of the liquid was observed after ten or fifteen minutes, and of the residual fat after the ether had evaporated, i. e., next day. The results obtained on nine genuine and seven process butters and two oleomargarines are given in Table X:

TABLE X.—*Results of "creeping test."*

Sample.	Height of creeping at end of 15 minutes.	Height of creeping after standing over night.
Process, A 22608	One-half inch	1 inch.
Dairy, B 22609	Remained level	Level.
Process, 23142	One-half inch	1 inch.
Creamery, 23143	Remained level	Level.
Process, 23144	One-half inch	1 inch.
Creamery, 23145	Remained level	Level.
	Height of creeping at end of 10 to 15 minutes.	Height of creeping after standing one hour.
Dairy, 22540	Level	Level.
Dairy, 22544	do	Do.
Dairy, 22545	do	Do.
Dairy, 22546	do	Do.
Oleomargarine, 22548	One-half inch	Three-fourths inch.
Process, 22555	do	One-half inch.
Process, 22556	do	Three-fourths inch.
Creamery, 22559	Level	Level.
Process, 22560	One-half inch	One-half to three-fourths inch.
Process, 22562	do	Three-fourths to 1 inch.
Creamery, 22564	Level	Level.
Oleomargarine	One-half inch	One-half to three-fourths inch.

These results marked so clearly the difference between the genuine and the process butters tested that we had great hopes this would prove an "absolute" test, but upon applying it to a series of canned butters from the Tropics the results obtained were so out of harmony with the indications of the boiling test that we lost confidence in it. Some unknown factor seemed to influence the results. Unfortunately lack of time prevented further study of the test. It is certainly worthy of careful investigation.

VII. THE WATERHOUSE TEST.

This test, first proposed as a test for oleomargarine, was made public at the December, 1900, meeting of the American Chemical Society by Prof. C. L. Parsons, Mr. C. H. Waterhouse, the originator of the test, having died some months before, after having communicated the test to Professor Parsons.

Through the courtesy of the last-named gentleman the writer was enabled to study the test some weeks prior to its appearance in the journal of the society,^a and the results of this study have been published in brief in *Farmers' Bulletin No. 131*,

^aMarch number, 1901, p. 200.

United States Department of Agriculture. As there stated, it was found that not only does oleomargarine respond to the test, but also process butter, and even some samples of genuine butter (apparently those that have been more or less melted), unless the temperatures of the test be carefully regulated.

Butter fat solidifies so much more quickly when cooled to a temperature below its hardening point than does oleomargarine that, being kept well broken up by the stirring required in the test, it will, if cooled rapidly enough, solidify in small granules, while oleomargarine will gather in a soft lump, unless the cooling be so very rapid and extreme as to granulate even the oleomargarine. But if the cooling be less rapid (by cooling with water less cold), then, under the conditions just supposed, process butter, or butter fat separated from the curd of butter, will also gather in a soft lump instead of granulating, and some genuine butters will do the same—apparently those that have been more or less melted. It may be that there are other causes for this, such as a scant amount of curd in the butter, but we have not yet had time to study this point.

In the original Waterhouse test milk is the medium in which the melted fat floats while it is cooled and stirred. Dr. Wiley suggested the use of water, and in all of our later trials we have used that medium.

It is probable that some genuine butters of the doubtful class would not behave the same in water as in milk, because of the fat of the latter, and perhaps also because of its proteid matter.

Our mode of operating the test is as follows: 100 cc distilled water in a small tin cup, $2\frac{3}{4}$ inches in diameter, are heated to just below 50° C, a slightly rounded teaspoonful of the butter is added, and gently stirred until melted; the temperature is then raised to exactly 50° , the cup is immediately placed in a pan 9 to $9\frac{1}{2}$ inches in diameter at the base, containing 1,000 cc of water at 12° C. The contents of the cup are then stirred rapidly with a wooden rod (more slender than a match) for 10 minutes with a circular and cross-wise motion in turn, in such manner as to keep the fat well agitated. The water in the pan is thoroughly mixed once every minute, the cup being used as a stirrer. At the end of 10 minutes' stirring the test is finished. A butter that is found to be granular (it may be so finely granular as to appear smooth) is above suspicion of having been "processed." Such, at least, is the result of our experience to the present time. A butter which gathers in a lump must be examined by other methods to decide whether it be "process" or not.

The Waterhouse test, applied as here described, is therefore useful as a means of proving whether or not a butter is surely genuine.^a

In conclusion I wish to give credit to my two assistants, Mr. J. H. Van Dyke, and Mr. D. Stuart, for their faithful and efficient aid in the work here recorded.

THE PRESIDENT. Those who are interested in butter certainly can not help being gratified that the Department has taken this matter up in such a thorough manner, at least pointing out and making clear some of the difficulties that have to be met.

We have reached the hour which was specially set aside for the report of the committee on nominations, which will now be in order, and will be presented by Mr. Huston.

^a We have recently encountered (April, 1902) two "straight" oleomargarines, made in Holland, that granulate, instead of gathering, in this test—a most surprising result, as yet unexplained.

REPORT OF COMMITTEE ON NOMINATIONS.

Your committee on nominations presents the following names:

For President, H. J. Wheeler; for vice-president, R. J. Davidson; for secretary, H. W. Wiley; for additional members of the executive committee, C. G. Hopkins, of Illinois, and F. D. Fuller, of New York.

On motion of Mr. Frear the report was received and the chairman of the committee on nominations, in the absence of the secretary, was instructed to cast the ballot of the association for the persons nominated. The chairman of the committee cast the ballot, and the persons named were declared duly elected as officers for the coming year.

The PRESIDENT. Next in order will be the report on tannin.

REPORT ON TANNIN.

By W. K. ALSOP, *Referee*.

The referee sent samples of chestnut extract, solid quebracho extract, vienna hide powder (marked No. 1), and dry chromed hide powder (marked No. 2), to fifteen chemists, requesting that analyses be made by the official method, and also after the following directions:

QUANTITY OF MATERIAL.

Use such quantity as will give 0.35 to 0.45 gram tanning material per 100 cc. Dissolve as in official method.

NONTANNINS.

(1) Prepare 20 grams of hide powder (No. 1) by digesting three days with 500 cc water, adding on each day 0.2 gram of chrome alum in solution. Wash by squeezing through linen, continue washing until no precipitate is obtained in wash water with barium chlorid. Squeeze hide in press and proceed exactly as in official method.

(2) To 14 grams of dry chromed hide powder (No. 2) in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently. Shake on shaker 15 minutes, throw on funnel with cotton plug in stem; evaporate 50 cc.

Determinations were requested on both dilutions of the extracts with wet chromed hide, and on the weaker solution by dry chromed hide.

Mr. Geo. P. Craighill last spring called the referee's attention to a method of analysis involving the use of chromed hide powder. He is using it in his laboratory with very satisfactory results. It is based on the method proposed by Mr. B. Weiss, of Vienna. As stated in an article in the *Leather Trades Review*, the essential details of his method are as follows:

To every 100 grams hide in 2 liters of water are added 3 grams chrome alum in solution, this alum solution being added in equal parts on three successive days. The hide is then washed until free from sulphates. To the last wash water some formalin is added. The hide is then pressed slightly and kept in a damp chamber.

Mr. Weiss states that he has kept it in that manner as long as two months, without decomposition or other change.

The nontannin determination is carried out as follows: To 130 cc of tanning solution (0.6 to 0.8 gram solids per 100 cc) add an equivalent of 7 grams dry hide, stir frequently, let stand overnight, squeeze through muslin filter, and evaporate 100 cc.

Mr. Craighill modifies the method as follows: After chroming he keeps the hide in water to which some formalin has been added, squeezing enough hide for each determination, then he proceeds as in the official method, using shaker. He also

states that he has kept hide some time in water with formalin in it without any change, but that he found hide deteriorated when kept as described by Mr. Weiss.

Mr. Craighill further states that he has prepared a dry chromed hide powder that retains the power of absorbing tannin sufficiently for use in analyses. Mr. Weiss says that he was unable to prepare such a powder. Mr. Craighill proceeds as follows:

The washed chromed powder is lightly squeezed, then thoroughly broken up and washed with alcohol, squeezed, the washing repeated, squeezed again and carefully "woolled," so that no lumps are left, then it is washed in ether, squeezed by hand and again carefully "woolled." The drying is done at ordinary temperature. It is essential that the finished product be entirely free from hard lumps, which condition can only be secured by extreme care during the process.

The dry chromed powder sent out with the samples was furnished by Mr. Craighill. It appears to be essential that hide so prepared be digested some time in the tannin solution and then shaken vigorously. This powder is free from solubles and by its use the correction for water introduced with the wet hide is avoided and the method simplified.

The most important claims for chromed hide, either wet or dry, are, that the soluble substances in the hide are rendered insoluble, and either for that reason or some other, not explained, it causes hide powders to give concordant results that otherwise differ widely by the method now in use.

This is an established fact, and for that reason alone the referee considers that he is justified in urging that the use of chromed hide be made official. A summary of the results of the cooperative work on samples follows.

TABLE 1.—*Chestnut extract—Official method (0.8 gram per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>	<i>Grams.</i>	
G. P. Craighill	53.69	46.48	44.68	1.80	15.86	28.82	No. 1, official.....	40.00	65.4	13.84	8 hours, 100° air bath.
					15.19	29.49	No. 1, chromed.....	38.00	64.4	13.53	Do.
					14.76	29.92	No. 2, dry chromed.....			14.00	Do.
H. T. Wilson	54.32	46.00	44.13	1.87	15.50	28.63	No. 1, official.....	36.00	64.68	12.70	Do.
					14.13	30.00	No. 1, chromed.....	44.20	71.92	12.40	Do.
					15.09	29.04	Laboratory, official.....	44.40	73.16	11.90	Do.
					13.38	30.75	Laboratory, chromed.....	42.00	68.45	13.25	Do.
H. C. Reed	54.11	45.58	44.79	.79	14.58	30.21	No. 1, official.....	40.40	68.60	12.68	8 hours, 105° air bath.
					14.02	30.77	No. 1, chromed.....	37.54	66.30	12.65	Do.
					14.07	30.72	Laboratory, chromed.....	34.44	65.70	11.81	Do.
F. P. Velch.....	54.51	45.36	44.39	.97	15.02	28.37	No. 1, official.....	70.00	80.60	13.58	4 to 7 hours, 70° in vacuo.
W. H. Krug	54.70	46.34	44.83	1.51	14.44	30.39	do.....				10 hours, 70° in vacuo.
					15.61	29.22	Laboratory, official.....				Do.
					14.10	30.73	Laboratory, chromed.....				Do.
F. H. Yocum.		46.08	45.57	.51	15.39	30.18	No. 1, official.....	50.00	76.4	11.80	24 hours, 96° steam oven.
					14.13	31.44	No. 1, chromed.....	50.00	71.7	14.15	Do.
F. H. Small	54.53	45.56	44.76	.80	14.65	30.11	No. 1, official.....	45.50	72.9	12.33	Do.
					14.03	30.73	No. 1, chromed.....	45.20	73.4	12.02	Do.
J. N. Hurty	53.19	46.77	45.70	1.07	16.77	28.93	No. 1, official.....	51.92	75.71	12.61	8 hours, 110°.
					15.90	29.80	No. 1, chromed.....	58.85	81.28	11.02	Do.
W. H. Teas.....		45.94	45.17	.77	14.00	31.17	No. 1, official.....	50.00	70.00	15.00	18 hours, 96° hot-water oven.
					13.80	31.87	No. 1, chromed.....	50.00	79.00	10.50	Do.
Kerr and Mosbaugh.....		48.04	44.42	3.62	15.71	28.71	No. 1, official.....				24 hours, hot-water oven.
M. S. McDowell.....		46.60	44.10	2.50	14.88	29.22	do.....	49.50			24 hours, 96° hot-water oven.
C. H. Brown		45.33	44.00	1.33	14.28	29.72	do.....	64.00	80.40	12.54	Do.
					14.06	29.94	No. 1, chromed.....	55.00	77.50	12.38	Do.
					14.11	29.89	No. 2, dry chromed.....			14.00	Do.

W. K. Alsop.....	45.66	44.22	1.44	14.58 14.41	29.64 29.81	No. 1, official..... No. 1, chromed.....	64.00 55.00	80.50 75.80	12.48 13.31	Do. Do.
Average.....	46.13	44.67	1.46	15.05 14.35	29.62 30.32	No. 1, official..... No. 1, chromed.....				
Highest.....	48.04	45.70	3.62	16.77	31.17	No. 1, official.....				
Lowest.....	45.33	44.00	.51	15.90	31.87	No. 1, chromed.....				
				14.00	28.37	No. 1, official.....				
				13.30	29.49	No. 1, chromed.....				
Greatest difference.....	2.71	1.70	3.11	2.77 2.60	2.80 2.38	No. 1, official..... No. 1, chromed.....				
Greatest difference from average.	2.38	1.03	2.16	1.72 1.55	1.55 1.55	No. 1, official..... No. 1, chromed.....				

TABLE 2.—*Chestnut extract—Experimental method (0.35 to 0.45 gram tanning material per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.		Water in wet hide.	Weight of dry hide.	Method of drying.
								Grams.	Per cent.			
G. P. Craighill	53.69	46.26	44.22	2.04	15.42	28.80	No. 1, official.....	40.00	68.4	68.4	12.64	8 hours, 100° air bath.
							No. 1, chromed.....	36.00	64.4			
H. T. Wilson	54.32	45.63	44.34	1.29	14.52	29.72	No. 2, dry chromed.....	36.55	64.46	64.46	12.99	Do.
							No. 1, official.....	41.00	69.85			
							No. 1, chromed.....					
							No. 2, dry chromed.....					
							do.....					
							No. 3, laboratory, official.	48.00	72.31			
H. C. Reed	54.11	45.75	44.95	.80	13.63	30.71	Laboratory, chromed.....	40.00	68.45	68.45	12.62	Do.
							Laboratory, official.....	45.90	72.2			
F. P. Veitch	54.51	45.59	44.83	.78	13.97	30.98	No. 1, chromed.....	38.33	69.1	69.1	11.85	Do.
							No. 2, dry chromed.....					
F. H. Small	54.77	45.52	44.41	1.11	14.00	30.95	No. 1, official.....	70.00	80.6	80.6	13.58	4 to 7 hours, 70° in vacuo
							No. 2, dry chromed.....					
F. H. Yocum	52.96	47.04	46.41	.63	15.27	29.56	No. 1, official.....	45.4	72.9	72.9	12.30	24 hours, 96° steam bath
							Laboratory, official.....	50.0	78.8			
M. S. McDowell	46.13	46.13	44.07	2.06	14.01	30.40	No. 1, chromed.....	44.7	73.4	73.4	11.89	Do.
							Laboratory, chromed.....	45.0	75.0			
W. H. Teas	46.00	46.00	44.86	1.14	14.92	29.49	No. 2, dry chromed.....				14.00	Do.
							No. 1, official.....		76.4			
M. S. McDowell	46.13	46.13	44.07	2.06	15.73	30.68	No. 1, chromed.....	51.0	71.7	71.7		24 hours, hot-water oven.
							No. 1, official.....	50.0				
W. H. Teas	46.00	46.00	44.86	1.14	13.88	30.69	No. 1, chromed.....	50.0			15.00	18 hours, hot-water oven.
							No. 1, official.....	50.0	70.0			
W. H. Teas	46.00	46.00	44.86	1.14	14.37	30.81	No. 1, chromed.....	50.0	79.0	79.0	10.50	Do.
							No. 1, official.....	50.0				

C. H. Brown	45.37	44.00	1.37	14.47	29.53	No. 1, official.....	64.0	80.40	12.54	24 hours, 96° hot-water oven.
				13.83	30.17	No. 1, chromed.....	55.0	77.5	12.88	Do.
W. K. Alsop.....	45.59	45.00	.59	14.18	29.82	No. 2, dry chromed			14.00	Do.
				14.46	30.54	No. 1, official.....	64.0	80.50	12.48	Do.
				14.01	30.99	No. 1, chromed.....	55.0	75.80	13.31	Do.
				14.06	30.94	No. 2, dry chromed			14.00	Do.
Average	45.88	44.70	1.17	14.74	29.96	No. 1, official.....				
				14.15	30.55	No. 1, chromed.....				
				14.43	30.27	No. 2, dry chromed				
Highest	47.04	46.41	2.06	15.73	30.81	No. 1, official.....				
				14.52	31.89	No. 1, chromed.....				
				15.72	30.95	No. 2, dry chromed				
Lowest	45.37	44.00	.59	13.38	28.80	No. 1, official.....				
				13.76	29.70	No. 1, chromed.....				
				13.99	29.11	No. 2, dry chromed				
Greatest difference	1.67	2.41	1.47	2.35	2.01	No. 1, official.....				
				.76	2.19	No. 1, chromed.....				
				1.74	1.84	No. 2, dry chromed				
Greatest difference	1.16	1.71	.89	1.36	1.16	No. 1, official.....				
from average.				.89	1.34	No. 1, chromed.....				
				1.29	1.18	No. 2, dry chromed				

*100 cc of solution used.

TABLE 3.—*Quebracho extract—Official method (0.8 gram solids per 100 cc.).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.	Used in filtering soluble solids.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>	<i>Grams.</i>		
G. P. Craighill	13.72	86.37	77.52	8.85	9.23	68.29	No. 1, official	40.00	65.40	13.84	8 hours, 100° air bath.	No kaolin.
					8.43	69.09	No. 1, chromed ...	38.00	64.40	13.53	do	
					8.38	69.14	No. 2, dry chromed ..			14.00	do	
H. T. Wilson	16.52	82.60	74.93	7.67	9.82	65.11	No. 1, official	46.00	68.48	14.50	do	Barytes.
					8.94	65.99	No. 1, chromed ...	44.00	74.59	11.17	do	
					9.04	65.89	Laboratory, official.	45.00	66.05	15.27	do	
					8.90	66.03	Laboratory, chromed.	45.00	66.46	15.09	do	
H. C. Reed	14.97	84.75	74.95	9.80	8.26	66.69	No. 1, official	41.13	70.20	12.30	8 hours, 105° air bath.	10 grams kaolin.
					7.91	67.04	No. 1, chromed ...	39.17	67.20	12.84	do	
					8.47	66.48	Laboratory, chromed.	34.84	67.20	11.42	do	
F. P. Veitch	16.17	83.64	75.62	8.02	9.17	66.45	No. 1, official	70.00	80.60	13.58	4 to 7 hours, 70° in vacuo.	
					8.43	65.37	do	47.20	72.90	12.79	24 hours, 96° in steam oven.	Kaolin.
F. H. Small	16.38	83.85	73.80	10.05							do	
					7.98	65.82	No. 1, chromed ...	49.50	73.40	13.17	do	
W. H. Krug	16.20	85.00	76.22	8.78	7.90	68.32	No. 1, official				10 hours, 70° in vacuo.	
					9.51	66.71	Laboratory, official.				do	
					9.54	66.68	Laboratory, chromed.				do	
					9.88	65.54	No. 1, official	50.00	76.40	11.80	24 hours, 96° steam oven.	Kaolin.
F. H. Vocum	14.22	85.78	75.42	10.36							do	
					8.87	66.55	No. 1, chromed ...	50.00	71.70	14.15	do	
					10.81	63.25	No. 1, official	54.91	77.00	12.63	8 hours, 110°	
J. N. Hurty	15.67	84.16	74.06	10.10	10.83	63.23	No. 1, chromed ...	54.74	81.28	10.28	do	

Keri and Mosbaugh M. S. McDowell	14.22	85.78	71.26	14.52	10.08	61.18	No. 1, official	51.00	70.00	15.00	24 hours, hot-water oven.	No kaolin.
		84.32	77.37	6.95	9.65	67.72	do				18 hours, hot-water oven.	Kaolin.
W. H. Teas		85.43	73.12	12.31	8.57	64.55	do	50.00	79.00	10.50	do	
					7.83	65.29	No. 1, chromed	50.00	78.50	10.75	do	
C. H. Brown	14.92	84.60	73.24	11.36	8.72	64.48	L a b o r a t o r y, chromed.	50.00	80.40	12.54	24 hours, 96° hot-water oven.	10 grams kaolin.
					8.27	64.97	No. 1, official	64.00	77.50	12.38	do	
W. K. Alsop	14.87	84.84	74.48	10.36	9.44	65.04	No. 1, chromed	55.00	80.50	12.48	do	10 grams kaolin.
					8.86	65.62	No. 1, official	64.00	75.80	13.31	do	
Average		84.70	74.77	9.93	9.23	65.54	No. 1, chromed	55.00	77.50	12.38	do	
Highest		86.37	77.52	14.52	8.66	66.13	No. 1, official	55.00	80.50	12.48	do	
Lowest		82.60	71.26	6.95	10.81	68.82	No. 1, chromed	55.00	75.80	13.31	do	
Greatest difference.		3.77	6.26	7.57	10.83	69.09	No. 1, official	55.00	80.50	12.48	do	
Greatest difference from average.		2.10	3.51	4.59	7.83	63.23	No. 1, chromed	55.00	75.80	13.31	do	

TABLE 4.—*Quebracho extract—Experimental method (0.35 to 0.45 gram tanning material per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.	Used in filtering soluble solids.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>		<i>Grams.</i>	<i>Per cent.</i>	<i>Grams.</i>		
G. P. Craighill	13.72	86.45	77.26	9.19	9.09	68.17	No. 1, official	40.00	68.4	12.64	8 hours, 100° air bath.	No kaolin.
					8.27	68.99	No. 1, chromed ..	36.00	64.4	12.81	do	
					8.34	68.92	No. 2, dry chromed ..			14.00	do	
H. T. Wilson	16.52	82.91	76.15	6.76	10.75	65.40	No. 1, official	46.00	67.96	14.73	do	Barytes.
					8.51	67.64	No. 1, chromed ..	44.00	74.59	11.17	do	
					8.59	67.56	No. 2, dry chromed ..			* 7.00	do	
					8.56	67.59	do			* 7.00	do	
					9.11	67.04	Laboratory, official.	45.00	66.34	15.14	do	
					8.34	67.81	Laboratory, chromed.	45.00	66.46	15.09	do	
H. C. Reed	14.97	85.40	77.93	7.47	8.96	68.97	Laboratory, official.	47.62	73.4	12.66	8 hours, 105° air bath.	5 grams kaolin.
					8.39	69.54	No. 1, chromed ..	37.88	67.9	12.15	do	
					8.83	69.10	No. 2, dry chromed ..			14.00	do	
F. P. Veitch	16.17	83.71	77.92	5.79	9.63	68.29	No. 1, official	70.00	80.60	13.58	4 to 7 hours in vacuo, 70°.	
					11.00	66.92	No. 2, dry chromed ..			14.00	do	
					9.94	65.96	No. 1, official	50.00	76.40	11.80	24 hours steam bath, 96°.	Kaolin.
F. H. Yocum	13.90	86.10	75.90	10.20	9.01	66.89	No. 1, chromed ..	50.00	71.70	14.15	do	
					8.73	67.42	No. 1, official	46.40	72.90	12.57	do	Kaolin.
					8.31	67.84	No. 1, chromed ..	46.40	73.40	12.34	do	
F. H. Small	16.45	84.21	76.15	8.06	8.86	67.29	No. 2, dry chromed ..			14.00	do	
					8.56	67.59	Laboratory, official.	50.70	78.80	12.08	do	
					8.92	67.23	Laboratory, chromed.	45.00	75.00	11.25	do	

W. H. Teas.....	85.50	74.07	11.43	9.09	64.98	No. 1, official.....	50.00	70.00	15.00	1 st hours oven.dodo	hot-water	Kaolin.
				8.51	65.56	No. 1, chromed ...	50.00	79.00	10.50			
	85.41	73.94	11.47	8.61	65.33	Laboratory, chromed.	50.00	78.50	10.75			
M. S. McDowell.....	85.34			9.72		No. 1, official.....	50.00			24 hours oven.do	hot-water	
				9.00		No. 1, chromed ...	50.00					
C. H. Brown	83.88	74.77	9.11	9.23	65.54	No. 1, official.....	64.00	80.40	12.54	24 hours, 96° oven.do	hot-water	10 grams kaolin.
				8.42	66.35	No. 1, chromed ...	55.00	77.50	12.38			
				8.71	66.06	No. 2, dry chromed ...			14.00			
				9.45	65.92	No. 1, official.....	64.00	80.50	12.48			
W. K. Alsop.....	85.20	75.37	9.83	8.71	66.66	No. 1, chromed ...	55.00	75.80	13.31			10 grams kaolin.
				9.01	66.29	No. 2, dry chromed			14.00			
Average	84.76	75.95	8.81	9.51	66.46	No. 1, official.....						
				8.57	67.38	No. 1, chromed ...						
Highest	86.45	77.93	11.47	8.99	66.96	No. 2, dry chromed						
				10.75	68.29	No. 1, official.....						
				9.01	69.54	No. 1, chromed ...						
				11.00	69.10	No. 2, dry chromed						
Lowest	82.91	73.94	5.79	8.73	64.98	No. 1, official.....						
				8.27	65.56	No. 1, chromed ...						
				8.34	66.06	No. 2, dry chromed						
Greatest difference.	3.54	3.99	5.68	2.02	3.31	No. 1, official.....						
				.74	3.98	No. 1, chromed ...						
				2.66	3.04	No. 2, dry chromed						
Greatest difference from average.	1.85	2.01	3.02	1.24	1.83	No. 1, official.....						
				.44	2.16	No. 1, chromed ...						
				2.01	2.14	No. 2, dry chromed						

* 100 cc of solution used.

These reports indicate—

(1) That the results obtained by the use of wet chromed hide powder are more accurate than those with unchromed hide. This is especially so in the results reported as obtained by the use of the weaker solution.

Several chemists did not report results with the dry chromed powder. Two reported that they could not get satisfactory results with it. The reports received are encouraging and indicate that the subject should be thoroughly investigated.

(2) That more concordant results can be obtained by the use of the weaker solution.

(3) That the method of drying has not much influence on the tannin result, provided the determinations in each analysis are dried in the same manner.

(4) That the determination of soluble solids is the weakest point in the method, and that some method must be adopted that chemists will follow.

Mr. Wilson reports as follows regarding the use of chromed hide powder:

Two things are well established. Powders unlike in absorptive power become alike when properly chromed. It is possible to produce a dry chromed powder ready for use. In support of the latter statement the referee has distributed a powder which yields results of astonishing concordance. The method for its preparation is very simple, and one which powder makers may easily adopt. In the opinion of the writer, the association should take official cognizance of this feature and recommend its adoption by powder producers.

Mr. Wilson reports in regard to "unlike powders being made alike," as follows:

TABLE 5.—*Quebracho extract*.—(Solution 0.35 to 0.45 gram tannin per 100 cc.)

VARIOUS HIDE POWDERS, CHROMED AND UNCHROMED.*

Hide powder.	Volume solution shaken.	Wet hide.	Moisture.	Dry hide.	Dry hide per 100 cc solution.	Nontan- nin.
	cc.	Grams.	Per cent.	Grams.	Grams.	Per cent.
I, unchromed	200	46	67.96	14.73	7.37	10.75
III, unchromed	200	45	66.54	15.14	7.57	9.11
I, chromed	200	44	74.59	11.17	5.59	8.51
III, chromed	200	45	66.46	15.09	7.55	8.34
IV, chromed	100	25	54.36	11.40	11.40	8.23
I, chromed	200	40	74.59	10.16	5.08	8.56
Do.....	200	50	74.59	12.70	6.35	8.57
Do.....	200	60	74.59	15.24	7.62	8.57
III, chromed	200	40	71.40	11.44	5.72	8.30
Do.....	200	50	71.40	14.30	7.15	8.33
Do.....	200	60	71.40	17.16	8.58	8.33
II, dry chromed.....	100			7.00	7.00	8.59
Do.....	100			7.00	7.00	8.56
Average all chromed powders						8.44

*All dryings at 100° in air, eight hours.

Mr. Wilson further reports:

It may be mentioned that powder No. 4 was simply a piece of white hide from the tannery, dried, rasped, degreased with ether, and chromed in the usual way. It will be noticed in Table 5 that diverse powders, after chroming, not only furnish concordant results, but may be applied in various ways and various quantities without altering the results; a condition impossible with unchromed powder. The range from the average of all the chromed powders is only 0.21 per cent, or less than 2.5 per cent of the whole percentage.

Mr. Wilson calls attention to that part of the table showing results obtained by him on hides Nos. I and III, when varying quantities of hide powders were used per 200 cc of solution, and he states that "with progressively increasing hide, no decrease of nontannin occurs."

Mr. Small reports some results obtained with a poor hide powder. He says:

These, although not obtained by the official method, may be of interest because the hide powder used is of exceptionally poor quality, it being impossible to get results of any value with it in the raw state.

TABLE 6.—*Determination of nontannins (Small).*

	Chestnut extract. Nontannins.		Quebracho extract. Nontannins.	
	Chromed.	Unchromed.	Chromed.	Unchromed.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Poor hide	13.68	17.38	7.89	11.84
Hide No. 1.....	14.01	14.35	8.31	*8.73

* Official method.

Mr. Reed reports some analyses made with his laboratory hide, and also a hide marked "B" in the table. The nontannins only are tabulated.

TABLE 7.—*Determination of nontannins (Reed).*

	Unchromed.		Chromed.	
	B.	Laboratory.	B.	Laboratory.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A solid quebracho extract, 9½ grams per liter.....	14.50	13.24	12.45	12.21
A solid quebracho extract, 6 grams per liter.....	15.10	13.18	12.02	12.23
Chestnut extract, 17½ grams per liter	15.99		13.68	14.07
Chestnut extract, 13½ grams per liter	15.90	14.25	13.52	
Chestnut oak bark extract.....		22.24		21.38

Mr. Reed states:

I am of the opinion that if hide powder B will answer the requirements, after being chromed pretty nearly any hide will do, for B is about as poor a hide powder as I have ever handled. That the chroming of B hide makes it fit for the analyses is abundantly proved by the result. The more analyses I make with chromed hide, the better I am pleased with the method.

Mr. Craighill has sent me results obtained with various powders unchromed, wet chromed and dry chromed. In the table following, A, B, and C are three different hide powders, the determinations being made on the same solution. Nos. 1 to 5 are results obtained on different extracts.

TABLE 8.—*Determination of nontannins (Craighill).*

	Nontannins.		
	Unchromed.	Wet chromed.	Dry chromed.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
A	20.35	19.48	
B	24.24	19.52	
C	22.57	19.63	
1.....	20.44	18.89	18.84
		18.79	18.76
		18.77	18.84
2.....	17.61	16.73	16.74
3.....	17.73	16.52	16.76
4.....	17.68	16.76	16.71
5.....	16.75		15.54
			15.72
			15.70

TABLE 8.—*Determination of nontannins (Craighill)—Continued.*

EFFECT OF CHROMING ONE DAY AND THREE DAYS.

	Nontannins.		
	Unchromed.	Wet chromed.	Dry chromed.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
3 days		16.61	16.56
1 day		16.94	16.86

Mr. Yocum reports as follows on a sample of Freiberg hide powder. The dry-chromed hide was digested in the tannin solution overnight.

TABLE 9.—*Determination of nontannins in Freiberg hide powder (Yocum).*

	Nontannins.		
	Unchromed.	Wet-chromed.	Dry-chromed.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Chestnut extract		14.81	15.02
Quebracho extract	12.15	8.93	9.01

With hide No. 1, wet chromed, Mr. Yocum's results were, with quebracho extract, 8.87; chestnut extract, 14.52.

Mr. Hurty reports the following experiments with dry-chromed hide, which was prepared as follows:

Fifty grams of hide were digested three days, with 3 grams of chrome alum in solution, 1 gram being added each day, washed free from sulphates, squeezed, digested twice with 98° alcohol to remove water, then dried. In all trials the same extract was used. The tannin solution was 1 per cent, and 10 grams of hide were used with 150 cc of solution, 50 cc evaporated. Laboratory hide was used for filter and official methods. The figures are for nontannins.

TABLE 10.—*Comparative determinations of nontannins by three methods (Hurty).*

Samples.	Filter method.	Official method.	Dry chromed hide.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
First set	16.1	17.38	16.76
			16.35
			16.60
Second set ^a			15.62
			15.66
			15.72
Third set ^a			15.52
			15.66
			15.18
Samples.	Filter method.	Official method.	Dry "formal hide."
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Fourth set ^b			15.18
			15.10
			15.54

^a Dry chromed hide prepared as above, except digested six days.

^b Obtained with "formal hide."

The "formal hide" was prepared as follows: Fifty grams of the hide was washed twice with distilled water, then treated for twelve hours with 10 cc of 40 per cent formaldehyde in 1,000 cc of water, washed once, squeezed, and treated twice with alcohol.

Mr. Hurty used twice as much chrome alum as was used in the other experiments reported. It is evident from his results that prolonged chroming with this amount of chrome tends to decrease the amount of nontannins.

The referee submits the following results, obtained on six different samples of extract, in the course of routine work. Hide powder A is Vienna hide, and B is a powder that gives very unsatisfactory results with the official method when unchromed:

TABLE 11.—*Determination of nontannins (Alsop).*

Hide.	Uchromed.	Wet chromed.	Three grams chrome to 100 grams hide.
	<i>Per cent.</i>	<i>Per cent.</i>	
1. A	16.34	14.87	Chromed 3 days.
B	18.72	14.77	Do.
2. A	16.25	15.06	Chromed 24 hours.
B		15.06	Do.
3. A	18.26	15.85	Do.
B		16.10	Do.
4. A	16.76	15.79	Chromed 5 minutes on shaker.
B	19.31	15.89	Do.
5. A	16.54	15.59	Do.
B	19.28	15.84	Do.
6. A	13.53	12.45	Do.
B	15.27	12.40	Do.

These results, beyond showing that chroming brings these two hide powders into accord, are probably not of much value, as they are from different samples of extract.

Chroming on the shaker has given very satisfactory results with some hide powders, but others plump so much that they are very difficult to squeeze afterwards. The greatest objection to the method for the use of wet chromed hide, as outlined in directions sent to collaborators, is the length of time required to chrome the hide—that is, three days. The referee considers this a serious objection, as it will delay analyses unless hide can be kept satisfactorily when chromed. This method, at best, is an experiment.

Since sending out the samples experiments have been made in order to determine whether the time can not be shortened without disadvantage.

Mr. Brown's experiments along this line follow.

TABLE 12.—*Determination of nontannins with regard to time involved (Brown and Alsop).*

BROWN.

Hide powder.	Chestnut extract (0.8 gram solids per 100 cc).	Chestnut extract (0.4 gram tannins per 100 cc).	Quebracho extract (0.8 gram solids per 100 cc).	Quebracho extract (0.4 gram tannins per 100 cc).
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Vienna, unchromed.....	14.28	14.47	8.72	9.23
Freiberg, unchromed	16.54	16.78		10.71
No. B, unchromed	16.47	17.12		
Vienna, chromed 3 days	14.06	13.83	8.27	8.42
Freiberg, chromed 3 days.....	14.76	14.34	8.20	8.23
No. B, chromed 3 days.....	14.04	13.91		8.41
Vienna, chromed 1 day	14.01	14.02		8.44
Freiberg, chromed 1 day.....	14.71	14.62	8.14	8.46
No. B, chromed 1 day	14.02	13.92		8.27
Dry chromed	14.11	14.18		8.71
Average for chromed hide				8.42
Average for chromed hide, Mr. Wilson's table, No. 5				8.44

ALSOP.

Vienna, unchromed	14.46	
B, unchromed	16.69	
Vienna, chromed 3 days.....	14.01	
B, chromed 3 days.....	13.81	
Vienna, chromed 1 day	14.05	
B, chromed 1 day.....	13.80	

These results show that there is no necessity for chroming hide longer than one day, at least, for the three powders tried, and if any one has worse samples for the official method than B and Freiberg they are unfortunate. One or two chemists have expressed the opinion that three days' chroming is necessary, or at least better, but have not submitted any results that support their views.

In Table 8 the results by one day and three days' chroming show about 0.3 per cent lower nontannins for the longer period. Mr. Hurty gets lower results by long chroming with larger quantities of chrome. Assuming that Mr. Craighill's dry-chromed powder chromed one day is free from solubles, as well as the hide chromed three days by Mr. Hurty, who states that he tested for soluble hide in filtrate with negative results, what warrant is there for chroming any longer, even if the nontannins may be lowered? The great advantage of the hide powder method is that it approximates the conditions in the tannery.

Vienna hide has been considered the standard by most chemists, because until recently it gave concordant results and a nontannin filtrate free from tannin and almost free from soluble hide. No other hide powders that the referee knows of fulfill these conditions as well. Some differ widely from Vienna and almost always give higher nontannins. Chroming causes these powders to give concordant results with a somewhat lower nontannin in the case of Vienna hide when 3 grams chrome per 100 grams hide are used. The referee believes that the best way to chrome is the method that will bring concordance with the least variation from the standard now in use.

Mr. Craighill says that his experiments point to 3 grams per 100 grams of hide as the best amount to use. This bears out experiments of the referee. The referee does not believe that there will be any material difference in hides chromed one day

or three days with that amount of chrome alum, but if chemists use varying amounts of chrome, much of the benefit of the method will be lost.

The referee urges that the chroming of hide powder one day (twenty-four hours), with 3 grams of chrome alum per 100 grams of hide be made the official method. This method will not delay analyses to any extent. Hide started one afternoon will be ready the next day. It is generally most convenient to dissolve extracts in the afternoon and analyze the next day.

He also advocates a provisional method for dry chromed hide and recommends that it be carefully experimented with next year; also that an effort be made to interest hide-powder manufacturers to find out if it is possible to produce this material in marketable quantities of a satisfactory quality.

The referee has decided to advocate the adoption of 0.35 to 0.45 grams tannin per 100 cc as the official method for solution of tanning materials. A comparison of tables 1 to 4 seems to warrant this decision, which is scientifically correct, in theory at least. It is the method adopted by the European association and brings our methods that much nearer in accord. The majority of collaborators who expressed any opinion declared in favor of it. Prof. H. R. Procter states:

We have found in practice that the difficulty of choosing a quantity of material, of which the tannin will fall within these limits, to be largely an imaginary one.

In regard to the volume of solution to be evaporated for the various determinations the following comments have been made:

Mr. *Krug*: I am not favorably impressed with the use of 50 cc for evaporation. This volume of solution leaves very little limit for error, and only doubles the errors already present and so far unavoidable.

Mr. *Wilson*: The method for nontannins involves a residue too small when 50 cc is used for evaporation. In the writer's opinion, the method should state a range of weight for the residue for different materials. This can be accomplished by substituting 100 cc for 50 cc in some cases. It appears to the writer that for total and soluble solids the residue as now given need not be changed.

Mr. *Teas*: I do not approve of the weaker dilution, nor the use of 50 cc for evaporation, the errors being increased when so small quantities are to be weighed.

Mr. *Yocum*: I am not in favor of the use of the smaller volume, the unavoidable errors being increased by its use and the shorter time required for evaporation in no wise compensating for the greater errors introduced.

Mr. *Reed*: The error resulting from the evaporation of 50 cc is multiplied, whether this error occurs in pipetting or in weighing. Without doubt this is the only disadvantage. The error is proportionately larger in the case of the nontannins. To offset this we have the following advantages:

- (1) The time gained in the performance of the analysis.
- (2) Time saved in process of filtration.
- (3) The error from oxidation upon reduction of time in drying is lessened.

In my opinion, the advantages of using 50 cc more than offset the disadvantages.

Mr. *Small*: I am in favor of using 50 cc, it being my opinion that more accurate work can be done with these smaller quantities.

Mr. *Craighill*: I am unqualifiedly in favor of 50 cc for evaporation, believing it to be more accurate. The longer time required for drying the larger residues has objections, to say nothing of the point of decomposition.

Mr. Brown and the referee, when making analyses of the official samples, did each in duplicate; also evaporated two 50 cc portions for each determination from the same solution. The result of their work, which involved a large number of weighings, shows that no closer duplicate weights can be obtained than when 100 cc is used. The referee agrees with Mr. Yocum that the time gained in evaporation of the smaller volume does not at all compensate for the greater errors introduced. He does not remember seeing any figures to show that errors introduced by oxidation are lessened by the smaller volume.

Mr. Reed states as an advantage the time gained in process of filtration. He gives in his report the following experiment and its results:

Twenty-nine solutions of quebracho extract were tested, each as follows: Five grams kaolin added to portion each solution, stirred, and 150 cc passed through and

discarded. The 50 cc following (the fourth) and the two following portions (the fifth and sixth) were evaporated. The comparison was made in order to prove whether we were obtaining, as nearly as possible, the correct soluble solids by the evaporation of the fourth 50 cc according to the then official method.

Soluble solids in quebracho extracts from the evaporation of the fourth, fifth, and sixth 50 cc filtered portions of the same solutions after discarding the first 150 cc passing through.

Number of quebracho solutions used.	Number soluble solids tested.	Kaolin used, in grams.	50 cc portion evaporated.	Number instances giving highest per cent soluble solids.	Percentage of instances giving heaviest soluble solids.
29	29	5	Fourth	13	44.83
	29	5	Fifth	11	37.93
	29	5	Sixth	5	17.24

This record does not show as great uniformity as could be desired. The percentage is in favor of the fourth 50 cc, but not very markedly so. The result would have been of greater value if the time of contact had been taken in each instance.

These figures the referee considers an argument in favor of the evaporation of 100 cc, which lessens the error in the determination of soluble solids.

The method proposed by Mr. Wilson, involving the use of 100 cc for some nontannin solutions and 50 cc for the other determinations, does not seem advisable nor one likely to be followed if adopted.

Mr. Carr, in his report last year, said:

Owing to the sensitiveness of tanning materials to the heat employed in drying, it is believed that the use of volumes yielding residues of from 0.400 to 0.500 mg is preferable to volumes giving greater residues. It is believed that if, instead of 100 cc, 50 cc shall be used for total and soluble solids, the resultant weight may be dried to constant weight in less time and at less risk of decomposition or oxidation.

The method as modified by him also involves the use of weights for nontannins as low as 40 mg.

It is interesting in this connection to note that the time for drying was not shortened.

The quantity of material advocated by the referee for solution of tanning materials involves residues somewhat heavier than the above when 100 cc is used, but in his opinion 50 cc would cause residues too small for the best results, and therefore the use of 100 cc for all evaporations is advocated.

Mr. Krug reports the following experiments on various methods of drying:

TABLE 13.—*Determination of solids with various methods of drying.*

OFFICIAL QUEBRACHO EXTRACT.

Method of drying.	Total solids.			Soluble solids.		
	A.	B.	Mean.	A.	B.	Mean.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
10 hours in vacuo, 70° C	85.05	84.95	85.00	76.32	76.11	76.22
8 hours in air bath, 100° C	84.47	84.74	84.61	74.95	75.05	74.90
8 hours in air bath, 110° C	83.26	84.21	83.74	74.74	74.74	74.74
24 hours in water bath, 98° C	85.37	85.37	85.37	74.74	74.74	74.74

OFFICIAL CHESTNUT EXTRACT.

10 hours in vacuo, 70° C	46.17	46.51	46.34	44.69	44.97	44.83
8 hours in air bath, 100° C	47.94	48.06	48.00	47.14	47.09	47.12
8 hours in air bath, 110° C	44.80	46.17	45.49	43.77	45.14	44.46
24 hours in water bath, 98° C	46.06	46.80	46.43	44.12	44.17	44.15

On these results Mr. Krug makes the following comment:

The comparative moisture determinations show considerable variation, and I believe this to be due to some loss by oxidation when the determinations were made by drying in air. Drying at 110° is in almost every case unsatisfactory, giving wide variations, and this temperature is no doubt too high and causes decomposition.

Last year one method for drying was changed from eight hours at 100° to 103°, in air bath, to eight hours at 100° to 110°. No reasons are assigned for the change in temperature, and the referee knows of none that justify it.

The temperature obtainable in a hot-water oven does not exceed 100°. Therefore it seems that no method of drying should exceed 100° unless it is made the only one to be used. The referee believes that the time stated for drying in the method is in excess of that required for practical purposes, especially if the weaker solution be adopted as official.

The results on total solids in the reports received from collaborators vary so much when dried under the same conditions that any comparison of the results based on different methods of drying are not of much value.

In the report on tannin presented by J. H. Yocum in 1898 Mr. Eachus contributed some valuable results on this subject.^a

The greatest variation in per cent of tannin, in Mr. Eachus's table, 0.43 per cent, is between duplicates both dried eighteen hours. The experience of the referee is that prolonged drying causes greater variations in weights between duplicates, due, probably, to decomposition or oxidation. It also gives slightly lower weights in most cases, but does not cause any material difference in the percentage of tannin.

The following changes in the method are recommended: From eight hours at 100° to 110°, in air bath, to six hours at 100°, and from twenty-four hours to eight hours at the temperature of boiling water, which means practically a hot-water oven where the temperature is rarely above 98°. The referee has had no experience with drying extracts in vacuo, therefore can express no intelligent opinion as to the time required.

With the exception of two, the collaborators expressed no opinion on the question of filtration for soluble solids. Mr. Craighill expresses himself as opposed to the use of kaolin or any other assistant. In a report on the subject, from Mr. Reed, the following table is found:

TABLE 14.—*Influence of time of filtration and proportion of kaolin upon soluble solids (Reed).*

Solid quebracho extract.	Concentration.	Kaolin.	Total solids.	Soluble solids.	Reds.	Volume of filtrate.	Time of contact.
	Grams in 100 cc.	Grams.	Per cent.	Per cent.	Per cent.	cc.	Minutes.
Analysis 1	9½	10	84.75	74.95	9.80	200	290
Analysis 2	9½	5	84.75	75.43	9.32	200	215
Analysis 3	9½	2½	84.75	75.73	9.02	200	150
Analysis 4	6	10	85.01	77.37	7.64	200	175
Analysis 5	6	5	85.40	77.93	7.47	200	135
Analysis 6	6	2½	85.40	78.37	7.03	200	120
Analysis 7	6	1½	85.40	78.70	6.70	200	110
Analysis 8	6	0	85.01	* 78.80	6.20	200	90

*Shows trace cloudiness.

Mr. Reed comments on these results as follows:

By comparing the time consumed in filtration for soluble solids in the case of the more diluted solution with that of the less diluted, it will be observed that the former

^aSee Bul. No. 56, Div. of Chem., Dept. of Agric., p. 106.

gives a much more rapid filtration than the latter when the amounts of kaolin are the same. At the same time the more dilute solution requires a less amount of kaolin to produce the requisite clear solution. Thus we have by employing a more diluted solution been able to reduce the amount of kaolin and the time of contact of the solution with the filter paper.

As the official method now stands for soluble solids "10 grams kaolin, previously washed in a portion of the solution, may be used when a clear filtrate can not otherwise be obtained." That this does not fully cover all possible contingencies must be true from the very fact that of two extracts of the same tanning wood one may give a clear filtrate without the use of kaolin while the other may require it. If we are comparing two such extracts for their relative tannin content, and in one case use no kaolin and in the other 10 grams of kaolin, as the official method indicates, we obtain results which are certainly not justly comparable; i. e., if we employ 10 grams of kaolin to obtain a clear filtrate, where a certain amount of kaolin is absolutely necessary, we should use 10 grams of kaolin for the solution where no kaolin is required. This must be true if with the same solution we obtain such widely divergent results in soluble solids with different proportions of kaolin. As stated by the referee in last year's report, "Absorption is largely a function of the time the solution is in contact with the paper," and this being true the addition of an equal amount of kaolin to a solution not of necessity requiring it will surely tend to make the time of contact more nearly the same as that of the solution requiring it. To recapitulate, it is the reporter's opinion that there should be some method suggested so that a uniformity in time of filtration for soluble solids could be brought about, taking into consideration the varying results with different proportions of kaolin as shown.

It may be stated that by the methods now in use clear filtrates can not be obtained with some extracts with any degree of certainty or within a reasonable time without the use of some assistant. In the absence of some better method, this association should either make no determination for reds, which is manifestly unjust, or it should adopt some method involving the least error that will insure a clear filtrate in all cases, and that method be followed for all extracts. The method as adopted last year advocated the use of barytes. Almost without exception chemists who have tried this method have discarded it. The following method is proposed:

To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, decant as much of the liquid as possible, add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc through, evaporate the next 100 cc. Double-pleated S. and S. No. 590, 15 cm filter paper shall be used.

Two grams of kaolin is sufficient to clarify extracts, therefore there is no need of using 10 grams, as the larger amount retards the filtration, and in addition introduces other errors. Although by no means perfect, this method has given good results, and does not materially affect results on an extract that can be filtered clear without kaolin.

The referee has made no experiments with the optional method for soluble solids, and therefore expresses no opinion about it.

The following modifications of the official method (Bulletin 46, revised, page 79, VIII, methods) for the analysis of tanning materials are recommended:

II. QUANTITY OF MATERIAL.—First line, read "give from 0.35 to 0.45 gram of tannins per 100 cc of solution." Sixth line, read "give from 0.35 to 0.45 gram of tannins per 100 cc of solution."

III. MOISTURE.—(b) 1. Read: "For eight hours at the temperature of boiling water in a steam bath."

(b) 2. Read: "For six hours at 100° in an air bath."

IV. TOTAL SOLIDS.—For 50 cc read 100 cc.

V. SOLUBLE SOLIDS.—Read: "Double-pleated filter paper (S. and S. No. 590, 15 cm) shall be used." Proceed as follows: To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, decant as much of the liquid as possible, add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc through, evaporate the next 100 cc.

VI. NONTANNINS.—Read: "Prepare 20 grams of hide powder by digesting twenty-four hours with 500 cc of water, adding 0.6 gram of chrome alum in solution. This solution to be added, one-half at the beginning and the other half at least six hours

before the end of the period. Wash by squeezing through linen; continue washing until the wash water does not show any precipitate with barium chlorid. Remove as much water, etc."

The remaining operations are identical with the official method, except that "100 cc" is read for "50 cc."

To VI add the following optional method:

To 14 grams of dry chromed hide powder in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently; shake on shaker fifteen minutes; throw on funnel with cotton plug in stem; let drain; tamp down the hide in funnel; return filtrate; filter until clear; evaporate 100 cc.

The PRESIDENT. Are there any papers on tannin?

Mr. KRUG. In connection with this subject I would like to address the association for a few moments in regard to the work that was done at the last meeting of the International Association of Leather Trades Chemists.

REPORT ON MEETING OF THE INTERNATIONAL ASSOCIATION OF LEATHER TRADES CHEMISTS, 1901.

By W. H. KRUG.

The International Association of Leather Trades Chemists met at Liège, Belgium, on August 28, 1901. The changes made in the method used by that association were as follows:

(1) The adoption of Freiberg hide powder, the maximum cellulose content to be 20 per cent, and the manufacturer to mark each delivery with the percentage it contains.

(2) The adoption of the A. O. A. C. method for used tanyard liquors.

Professor Procter formally proposed the adoption of the Association of Official Agricultural Chemists' method as official, but the motion was lost, the comparative results obtained by the members indicating that it was not more exact than the filter-tube method. The subject of filtration was discussed at length, and the association was forced to the conclusion that in the determination of the soluble solids a perfectly clear filtrate is absolutely necessary. The members expressed much dissatisfaction with paper No. 605, owing to the fact that it does not give uniform results and the filtration is exceedingly slow, but no change was made pending experiments which are to be reported at the next meeting. In connection with the adoption of Freiberg hide powder a subcommittee was named which will formulate methods for testing the suitability of various hide powders and for determining the moisture, ash, cellulose, and nitrogen in them. Dr. Paessler presented a series of results obtained with chromed hide powder and showed that it possesses no special advantages. This was confirmed by Professor Procter and others. The difficulties encountered in the analysis of extracts which have been decolorized by the addition of bisulphites was discussed and it was shown that the addition of a trace of hydrochloric acid to the solution gave results from 2 to 4 per cent higher than by the ordinary method. No definite conclusion was reached by the association, Professor Procter stating that the present rules permitted the addition of acid, provided this was stated in the report.

Professor Procter presented a lengthy report dealing with the differences which to-day exist between the International and Association of Official Agricultural Chemists' methods. With a considerable number of comparative experiments as a basis he arrived at the following conclusions:

(1) That rapid cooling of the diluted extract on the whole gives a somewhat higher result for soluble solids than slow cooling, and, furthermore, a smaller mean

error, so that the advantage appears to lie with the International method, which also has the merit of rapidity and convenience.

(2) That flat-bottomed dishes possess no advantage over round-bottomed dishes, the residues drying as rapidly in the latter as in the former.

(3) That the variations, which American chemists claim to be caused by the method of cooling, are more probably due to the greater oxidation which attends the longer period of drying prescribed by the Association of Official Agricultural Chemists' method.

(4) That errors may often be caused by inaccuracy of the weights used.

(5) That the determination of the soluble solids must be made on an absolutely clear filtrate to obtain concordant results, the kaolin filtration being probably the best method known for this purpose.

(6) That the compulsory adoption of an expensive quantitative paper like S. & S. No. 590 is not warranted, it being probable that any paper free from soluble matter will be fully satisfactory when kaolin is used.

(7) That the filter-tube method gives the lowest result in nontannins, the Association of Official Agricultural Chemists' method averaging from 2 to 3 per cent higher and the accuracy being somewhat greater with the former, although in both cases the mean error may be neglected.

(8) That the Association of Official Agricultural Chemists' method is less affected by differences in the hide powder, especially when the powder is faintly acid.

As previously stated, the association decided not to abandon the filter-tube method, though the Association of Official Agricultural Chemists' method was adopted for spent liquors.

The question of using a chromed hide powder has occupied the attention of American chemists for some time, and it may be of interest to quote from Professor Procter's report.

Experiments have been made on the use of chromed hide powder, as proposed recently by Weiss, though they are not so exhaustive as might be desired. Powder was chromed in several ways, using a basic solution of chrome alum, added slowly over three days, and also by drumming for half an hour * * * but none of these gave as good results as that chromed according to Weiss's original directions.

Weiss's statement was confirmed that the powder became less absorbent when strongly pressed, a lot of powder accidentally pressed to 30 per cent of water giving with chestnut extract and Weiss's method of digestion overnight a result of 10.1 of nontannins, as compared to 7.1 by the same method when the water was reduced to only 90 per cent. With the same extract the filter method showed only 6.3 per cent. It was found that shaking by the machine for 20 minutes gave somewhat lower results in nontannins than standing overnight. In each case powder equal to 7 grams of dry powder per 100 cc was employed.

It was found that while the powder with 30 per cent of moisture was quite sweet and good after three months, that with 90 per cent was putrid in a week. I am far from saying that this use of chromed hide powder is not worth further experiment, but it does not seem to have reached a completeness which would justify us in considering it as an official method, and where the shaking machine is used it is more troublesome and probably not more exact than untreated hide powder. It is also most probable that the presence of chrome and formaldehyde would alter the ratio of absorption between different tanning materials at least to some extent.

In conclusion, I desire to read a letter recently received from Professor Procter and would suggest that the method of determining the absorption—correction for the filter paper in the determination of the soluble solids described therein—be made the subject of an investigation by the referee for the ensuing year. The letter is as follows:

LEATHER INDUSTRIES LABORATORIES, THE YORKSHIRE COLLEGE,
Leeds, October 25, 1901.

DEAR MR. WILEY: We are experimenting here with a method for correcting the results of filtration of tanning liquors through different papers, which in view of the early meeting of the Association of Official Agricultural Chemists I hasten to communicate to you, although we have not yet tested it at all fully. The principle of the correction is that we adopt such a method of filtration in the first instance as will give a perfectly clear filtrate with the material in question. Suppose, for instance, that to do so we employ paper 590 and 3 grams of kaolin. In order to establish the correction for the particular material and for this special mode of filtration we make

a second filtration, using double paper 590 and 6 grams of kaolin. Naturally this work must be done at least in duplicate to avoid experimental errors. In the second filtration we have practically double the true absorption which takes place in the first, and consequently the second liquor will leave a smaller residue than the first one. The difference between the two, then, is the correction required, and must be added to the result with the single filtration in order to compensate for the loss with 1 paper and 3 grams of kaolin. Of course, there is the theoretical objection that the liquor on passing through the second paper is already weaker and presumably gives up somewhat less tannin to the paper, but a little numerical consideration will show that this correction of a correction is quite too small to influence the results of analysis. The method is easy of execution, the filtration through double papers taking but little longer than that through a single one, and from the work we have already done it is evident that the results if not absolutely accurate at least very much diminish the error which the paper introduces. Of course, if this should turn out on practical experience to be the case it at once unifies our methods with regard to filtration, since for any particular extract we shall use the paper we find most satisfactory and make the corresponding correction, of which in many cases one determination will serve for a whole series of analyses, the whole thing being only a question of tenths per cent.

At the meeting of the International Association in Liège the absolute necessity of filtering to perfect clearness was strongly insisted upon, and I very fully indorse this as being an essential to concordant results. Of course, it may happen that in some cases a tanning material contains small quantities of a coloring matter or some special substance which is fully absorbed by the first paper, and in this case that substance must necessarily be reckoned as insoluble matter. Whether these cases ever arise is uncertain, but some conclusion may be arrived at by experimenting with extract infusions and liquors which are optically clear without filtration. In this case the whole loss should take place in the first filtration and no loss or a much smaller one in the second. I have not, however, yet come upon any cases which support this idea.

Hoping that some of your chemists will take up the matter and confirm or condemn the method which I suggest,

I am yours, faithfully,

HENRY R. PROCTER.

The PRESIDENT. Professor Atwater asked to have a statement made on the floor which might be of interest to some individuals in the association, and Mr. Woods, I believe, was asked to make the statement. Mr. Woods has the floor.

Mr. WOODS. Of course you know the character of work Professor Atwater is doing along the line of nutrition investigations. He needs one or two men to cooperate with him in connection with that work. I do not suppose the salary would be very large, possibly \$1,000 or \$1,200. If there are any young men here interested along that line of work—the nutrition of man—I suggest that they write to Professor Atwater.

Mr. HOPKINS. I think it would be well to have Mr. Woods make a similar announcement for several others of us here. We are looking for men.

The PRESIDENT. We might open an exchange department.

Mr. WHEELER. I would like to make a similar announcement in connection with the Rhode Island station.

The PRESIDENT. We will proceed now to receiving the reports of the committees on recommendations of referees. First, Mr. Huston will report for Committee A.

REPORT OF COMMITTEE A ON RECOMMENDATIONS.

Committee A has to report that there are very few radical recommendations submitted. The first thing that has been called to the attention of the committee is a request to have the method of making standard hydrochloric acid corrected in the methods. I think if the secretary will take a note of it he can correct it himself. The error is plain enough when attention is called to it.

NITROGEN.

The first recommendation is that the neutral permanganate method be adopted as a provisional method. Your committee suggests that we change the phraseology and say that it be printed as a provisional method.

The second recommendation is that the alkaline permanganate method be further tested by the succeeding referee. I would move the adoption of these two suggestions.

Carried.

There is a third recommendation which is not clear to the committee. It reads:

That a caution be inserted in the method for total nitrogen concerning the proper digestion bath of sulphuric acid with certain classes of material, or that the question be investigated by the referee.

In examining the report the only thing we can find to which this recommendation seems to refer is that sometimes the digestion is stopped too early. I believe, since it is in a form which is not at all clear, it would be well not to take any action upon this third recommendation.

POTASH.

There are two recommendations under this head.

First. That the causes of low results obtained by the official method be investigated. This relates particularly to high-grade potash salt. Second. That the milk-of-lime method be submitted for investigation for the ensuing year. I move the adoption of these two recommendations.

Carried.

There is another matter in connection with potash which has been brought to our attention by Mr. Von Herff; that is, that the method for determining the water as laid down in our official methods is entirely different from that used by the German Kali Company. I do not know that any action is necessary beyond suggesting that the referee consider the matter and make some report next year. The Kali Company heat to a low red heat, which of course is very different from our procedure.

PHOSPHORIC ACID.

There is no formal recommendation in this report. Under the head of recommendations there is a discussion as to whether it might not be well to take up the question of the availability of the phosphoric acid of basic slag, and a suggestion that the method of digestion with citric acid would be a proper basis upon which to proceed. There is, however, no formal recommendation on this, so I presume the referee will be at liberty to take up such work as he wishes next year.

ASH.

(1) It is recommended that the title of this section be changed from "Methods for the analysis of ashes" to "Methods for the determination of inorganic plant constituents." The reason is that there is no method known, at least at present, by which a plant can be burned and all the sulphur, chlorine, and other constituents

left in the ash. That is to say, we are not able to prepare an ash which contains all the inorganic constituents of the plant. I move the adoption of this recommendation.

Carried.

(2) It is recommended that the method for the determination of sulphur in plants, called the nitric-acid method, as modified, be adopted as a provisional method. The committee has taken the liberty of changing phraseology a little. There has been absolutely no change in the subject-matter:

Place 5 grams of material in a 3½-inch porcelain evaporating dish, add 20 cc of nitric acid (conc.), and heat the mixture cautiously on a water bath until all danger of overflowing is passed. Partly evaporate, add 10 cc of a 5 per cent solution of potassium nitrate, evaporate the mixture to complete dryness, and ignite, at first gently and then under a blast lamp, until the residue is white. Then dissolve in hydrochloric acid, evaporate to dryness, and heat for some time in an air bath to render silica insoluble. Take up the residue in water, with the addition of a little acid, filter, and precipitate the sulphuric acid with barium sulphate, etc., in the usual way.

I would like to ask about the following phrase: "At first gently and then under a blast lamp." The committee is in doubt as to whether it is intended that the ignition be made, as in some of the steel works, by inverting a blast lamp or not.

Mr. FRAPS. I would say make it over a blast lamp.

Mr. HUSTON. I move the adoption of this recommendation.

Mr. FREAR. May I inquire whether that is a recommendation of a provisional method?

Mr. HUSTON. Yes; entirely provisional; not official at all.

The recommendation was adopted.

Mr. HUSTON (resuming report of Committee A):

(3) It is recommended that the determination of chlorin be omitted from the methods until a method can be adopted which will give something like the truth.

Of course this refers to the method after the section is renamed. I do not understand that there is any particular fault found with the determination of chlorin in ash. The fault is with the determination of chlorin in the plant. I therefore move the adoption of this recommendation.

Adopted.

(4) It is recommended that the determination of potash by ignition of the plant substance with sulphuric acid, as described in the determination of potash in fertilizers, be adopted as an alternative method. I move the adoption of this recommendation.

Adopted.

The entire method of analysis has been rewritten in the report on ash, and as we had no copy of our methods here with which to compare them, the committee virtually decided that the methods as rewritten by the referee on ash be transmitted to the secretary for incorporation in the report and recommended for adoption. I therefore make a motion to that effect.

Carried.

Mr. VEITCH. Did the committee consider the use of the Eschka method for the determination of sulphur at all? It was spoken of in the meeting yesterday.

Mr. HUSTON. We did not consider that method; but, of course, at the time the referee's report is under consideration here it is quite in order for any member to move that the referee be instructed to consider that method next year.

Mr. VEITCH. I move that the referee be instructed to consider the Eschka method for the determination of sulphur in plants.

Carried.

SOILS.

There are no formal recommendations in connection with the report on soils; that is, in regard to the matter of soil analysis. There is, however, a recommendation that a committee be appointed to take up the subject of soil sampling, and your committee on recommendations move that such a committee be appointed. I think a committee of three would be sufficient.

Carried.

Mr. HOPKINS. It seems to me that if such a thing can be done it would be well for us to try to arrive at some provisional method for sampling soils at this time. A method of sampling soils is not quite the same as laying down a method of analysis. There is a large amount of soil investigation at our own station that must be done during the coming year, and it is necessary that we at least follow some method, and I would like, for my part, some expression of opinion from the members of the association, and, if possible, that we might adopt a provisional method for sampling soils. I have written the following brief description of a method that we use, and regarding which I have talked to several members of the association.

METHOD FOR TAKING SAMPLES OF SOILS FOR ANALYSIS.

By C. G. HOPKINS.

All samples of soils taken for analysis should be composite and should be composed of representative samples taken from at least five different places in the field sampled, each individual sample to be a column of uniform soil extending through the stratum sampled.

One composite sample should be taken from each important and distinctly different soil stratum to a depth of 40 inches, or 1 meter, including a composite sample from the arable stratum, or plowed soil, usually about 6 inches or 15 cm. deep.

If the plow line and the subsoil line coincide, and the subsoil is a fairly uniform stratum to a depth of 40 inches, then only two composite samples need be taken, one of the arable soil and one of the subsoil. But if the subsoil line is lower than the plow line and not below 40 inches, then both strata below the arable soil should be sampled, which would make three strata to be sampled and necessitate the taking of three composite samples from the field—one from the surface or arable soil; one from the subsurface soil, that is, from the stratum between the plow line and the true subsoil line, and one from the true subsoil.

Mr. HOPKINS (continuing). I may say that the latter condition obtains in most of the soils in Illinois. We have the plow line about 6 to 7 inches from the surface, and we plow to about that depth. We have a soil that

is quite similar to the arable soil, a rich, black loam, that extends to 18 or 22 inches. Then we reach the subsoil line, and below that there is usually a stiff clay. If we go down 3 or 4 feet we have three distinct types of soil—certainly upon our old, worn land; that is, we have 6 or 7 inches which has perhaps been turned and turned and mixed for from forty to one hundred years; then we have, about 12 inches below that, soil which was once somewhat similar, perhaps rich, black loam, but which has never been disturbed and which may be very rich in some constituents that have been exhausted from the plowed soil; and from 20 inches to below we have usually a rich subsoil.

I would at least like an expression of opinion from those who are familiar with this subject, and see if it is not possible to adopt some provisional method; and I would move the adoption of this method as I have suggested, perhaps with some modifications or amendments.

MR. WILLARD. We are interested in soil more or less in Kansas, and I am very glad this question has come up. I have noticed the lack of a uniform method. I am very much pleased with what Mr. Hopkins has suggested here, and it is in line with what we have done. It seems to me that from the present light that is a very good provisional method. I second the motion.

MR. HUSTON. I think the method outlined by Mr. Hopkins has some very good points, and that it is very well adapted to the prairie, but I do not think it a good method to use in a section where there is rock. It seems to me that we had better discuss the subject in two parts. First, the sampling of surface soil, on which I think we can agree, and take up the subsoils separately. I believe a great deal in regard to subsoil will have to be left to the discretion of people in different sections of the country.

MR. HOPKINS. I did not mean to make any fixed regulation here for sampling the subsoil. I simply stated that representative samples should be taken from each important and distinctly different stratum. It may happen that there are three. It may happen that the plow line and the subsoil line will coincide, and in that case we would only have two samples. I have found soils where four or five different samples should be taken, because there were distinctly different strata.

The motion to adopt and print the method proposed as a provisional method was carried.

MR. HUSTON (resuming report of Committee A):

The matter of soil sampling has virtually, then, been settled. Another question which was brought up was the precipitation of the last trace of barium by the use of ammonium sulphate in determining potash. Your committee recommend that this be referred to the next referee on soils, and I make a motion to that effect.

Carried.

The attention of the committee is also called to the matter of methods for determining soluble portions of alkali soils, and, in general, any special methods necessary

for handling alkali soil; and we move that this matter be transmitted to the referee, and that he be asked to report at the next meeting what special methods may be needed, if any, in addition to our present method.

Carried.

The question of mechanical analysis of soils has been called to the attention of the committee. This has been before the association before more than once. While we recognize the importance of mechanical analysis of soils, we have not heretofore considered that a chemical subject; and while it is very closely related to our work, I think that we are hardly prepared to do more at present than to recommend that the referee take this matter up and be prepared to discuss it before the next meeting of the association. We make a motion to that effect.

Carried.

The following resolution was introduced:

Resolved, That the policy of the association as to its way of investigating analytical methods for the determination of available plant food be changed.

That in place of asking the cooperation of all chemists in the comparative study of two or more methods on a furnished set of samples, each of those chemists who are willing to participate in the work be asked by the referee to take up such a line of soil study by any method he may choose upon known soils of his own State or upon other known samples, the avowed purpose of such study being the working out of a chemical method for determining the available plant food of soils.

Further, that those who engage in this work shall keep the referee informed of their lines of work, shall furnish him with a summary of their results, to be presented by him to the association at the regular meetings, and shall from time to time, as the occasion warrants, present to the association the details of their work.

In regard to this resolution your committee believe that it is inspired by a proper motive, but we do not wish to limit the referee in his method of working. The referee has the authority to do his work in any way that he thinks best, and while we think this resolution ought to be transmitted to the referee as a suggestion or an aid in his work, we do not feel justified in moving its adoption as controlling the policy of the association or as limiting the referee, although, of course, any member of the association can move it if he wishes.

Mr. FREAR. I would move its reference to the referee on analysis of soils for his consideration.

Mr. HUSTON. This committee will second that recommendation.

Adopted.

Mr. HUSTON (resuming report).

INSECTICIDES.

The next is the report of Mr. Haywood, the associate referee on insecticides. He recommends that methods 1 and 2 be continued as provisional methods. These are the two methods of which he gave you the results this morning, which seem to be reasonably satisfactory, or the most satisfactory that have been used, and to warrant further investigation. We therefore move the adoption of this recommendation.

Carried.

The next recommendation is that of the other section of the committee on insecticides in particular relation to formalin and that class of material. This recommendation is to the effect that the methods be continued, with the omission of certain nonessentials in the testing work. There are some points in connection with this to which attention is called here in regard to testing potassium cyanid and materials of that nature, which the committee do not consider essential; and therefore they

recommend that the referees continue to test such materials, but that they confine their work to the essentials and limit it somewhat. We move the adoption of that recommendation.

Carried.

Mr. HUSTON. There is one other matter in connection with the report of this committee. I notice that nothing seems to have been done by anyone in testing formalin in which the cyanid process was used. The process must be some five or six years old, I should think; and perhaps one reason of its omission is that when it was translated by Allen it was very badly mutilated. The process is practically unintelligible as it stands in Allen, even in the revised edition. Some two years ago I had occasion to work on quite a number of samples of formalin and compared all the methods I could get hold of. This cyanid method seemed to be promising, and when I got the original account I found it worked out into a very serviceable, quick, and accurate method of determining formalin, very much better than any other method I could get hold of. I would like to suggest to the referee that that method be included. I can give him a description of the method, or he can get it from our annual report of last year. I believe it to be a very promising method for testing formaldehyde, and I would move that the referee be requested to look into the matter of the cyanid method for testing formaldehyde.

Mr. HAYWOOD. Before going into that, may I interrupt a minute to ask if Mr. Voorhees does not in his report call attention to the fact that formalin determination really belongs more to the food referee than it does to the referee on insecticides, and therefore he nearly goes to the length of recommending that it be entirely dropped by the referee on insecticides and referred to the referee on foods? They are carrying on nearly the same line of work.

Mr. HUSTON. The food committee, I think, are more particularly concerned in qualitative tests of formalin rather than the quantitative. I think they are hardly prepared to do any quantitative work on the small amount that is usually put in food. While attention is called in the report to the matter of which you speak, nevertheless the reporter afterwards recommends that they keep on testing formalin; so there is nothing for this committee to do but to present his recommendation.

The president brought up the question of whether this session should be continued until the work of the convention was finished, or whether it should adjourn and another session be held the following day. It being found that the hall would be required for a lecture at 4.30 it was decided to follow the latter course.

Mr. Wiley extended an invitation to the members to visit the laboratories of the Bureau of Chemistry of the Department of Agricul-

ture, and suggested that they do so when the meeting adjourned, as some of the men would be at the laboratory to receive them and the following day the building would be closed.

The president announced that a photographer was ready to take a picture of the members, and he asked that they assemble on the front steps immediately after adjournment.

Mr. Ross moved that the president-elect appoint during the sitting of the convention the committees on recommendations of referees who are to serve during the next annual convention.

The motion was referred to the committee on resolutions.

Mr. Ewell offered the following resolution and suggested that it be referred to the referee on nitrogen for the coming year to report a definite plan of subdivision:

Resolved, That the title "Determination of nitrogen" be changed to read "Determination of nitrogen and nitrogenous bodies," and that the work on nitrogen be divided between two or more referees. Further, that the nitrogen methods be taken out of fertilizer methods and proper reference inserted in their place.

At 4.15 p. m. the convention adjourned until 9.30 the next morning.

THIRD DAY.

SATURDAY—MORNING SESSION.

The meeting was called to order by the president at 10 o'clock.

The PRESIDENT: First in order will be the report on sugar, by Mr. Ewell.

REPORT ON SUGAR.

By E. E. EWELL, *Referee.*

I promise you in advance that I shall not read any long-winded report. The report on sugar this year, as last year, consists largely of an apology, and I feel that I owe an apology to the president for having made his administration in this regard lacking in success. I accepted the place with considerable hesitation, as I knew my time was very much filled up; but it was a subject of great interest to me, and I had hopes that I might be able to do something. I was compelled, however, to put it off from day to day until it was too late to resign with justice to anybody or to inaugurate any work that would be of any value to the association.

There are several important lines of work which ought to be taken up. It is gratifying to say, however, that they are not being neglected. This is a time when research work with regard to bringing out new methods is of more importance than the trial of methods which we now know to be lacking in certain regards. The original plan was that we should take up the methods of analysis of sugar beets alone. There is a great need of an official method, because there have been numerous disputes in the districts where beets are grown and sent to the factories on test.

You will recall that when I accepted the position of referee some years ago the subject was enlarged so as to include all soluble carbohydrates, at that time of importance in our work. I am thoroughly impressed with the idea that the change was a good one, but the only difficulty that has attended it was that there were no referees for the subheadings. That was one thing I had in mind when I asked for the amendment to the constitution which authorizes the executive committee to appoint a subreferee on such subjects when it deems wise.

The reports of the methods as now in the bulletin at the time of their last extensive revision represented very well the state of our knowledge of carbohydrates—that is, from the analytical standpoint. But much has been learned since then in regard to the methods and also as to the need of improvement in the methods. There is one very important need, and one which investigators have endeavored to supply in a measure, and that is one method of manipulation for determining all kinds of reducing sugars. It is greatly to be desired that some one shall spend most of his time for a year on this work; it would make a good year's work. I wish I could do it myself, but there is no prospect that I can.

I want to say a word in regard to the optical methods. The methods were changed just after I was referee, some years ago, and through some error—I do not know just how to explain it. I recommended that the old Clerget method and the Lindet

method be dropped and the method then known as the German method be substituted. This recommendation was based on the fact that all the research work previous to that time had been done with the German method. There is really no reason why that change should have been made, and there is every reason why the German method should have been retained and the present one discarded. But inasmuch as there is research work in progress now, seeking an inverting agent without action on other carbohydrates than sucrose, it does not seem wise to make any change until methods of that kind have been perfected. Mr. Tolman is actively working on such a method at this time. I shall be glad to have him speak on that subject himself, if he desires to do so.

I have written out two or three recommendations, which refer only to the division of the work. I recommend that there be a referee for the optical methods and a referee for the study of methods for the determination of reducing sugars, with the idea that if the referee can possibly arrange with some one—perhaps an instructor in a school—or with several, who can spend some time on this branch of analytical chemistry, the results may give the analyst something as a starting point. There should also be a third referee for the analytical methods of the sugar industry. I therefore recommend that the work on sugar be divided and that associate referees be appointed for three divisions of the work, as follows:

1. Optical methods.
2. Chemical methods.
3. Special analytical methods used in sugar industry.

The president referred these recommendations to the chairman of Committee B.

The PRESIDENT. Are there any papers, or any remarks?

Mr. ROSS. I have no special paper. I have a piece of apparatus that it will only take a minute or two to exhibit. It is used in a modification of the electrolytic process of determining invert sugars, or rather of determining the copper which is thrown down in this process. Quite a number of years ago there was a process proposed by Formanek in the *Zeitschrift für Rüben Zuckerind in Böhmen*, vol. 14, p. 178, and this, I think, was the first proposal that had been made to utilize the electrolytic method for estimating the copper thrown down from the Fehling solution by reducing sugars. In this original process the precipitate of copper suboxid was brought on an asbestos filter and washed thoroughly, then dissolved in 1.20 sp. gr. nitric acid, I believe, and the filter washed thoroughly to remove the copper from the asbestos, which was a very difficult undertaking. This solution, which should not contain more than 4 to 5 per cent of nitric acid, was electrolyzed, a large platinum dish being used for this solution. Other modifications of this method have been employed, involving the use of sulphuric acid and subsequent evaporation of the solution in order to expel the bulk of the nitric acid. A number of years ago I presented to this association a paper, which was also published in the *Journal of the American Chemical Society*, I believe, giving a modification of this method in which no attempt whatever was made to dissolve the precipitate on the asbestos. A large series of experiments which I made at that time showed that if you place the asbestos filter with the pre-

precipitate in a beaker, in contact with a platinum spiral, which was used as the anode, and if a 4 per cent nitric-acid solution was poured in and a platinum cylinder suspended in that, to act as a cathode, the copper suboxid would be oxidized in contact with the anode and would go thoroughly into solution just as the deposition took place on the cathode. In fact, the solution of the copper suboxid was always effected before the completion of the deposition occurred.

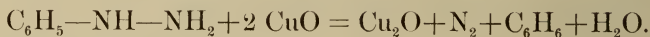
Now, the apparatus which I have here carries out this same principle, but in a little different way. Here we have a funnel tube. The original funnel tube that I had made for this purpose by Herman Kobe, in Berlin, was broken. This tube, which is almost an exact duplicate, is a funnel filter tube, and in the lower portion is a platinum spiral with one end melted through the glass. An asbestos filter is placed in this, filtration being accomplished by means of a pump, as usual. After washing is completed you turn off this stopcock, pour in a 4 per cent solution of nitric acid, insert the platinum cathode, make connections with the battery or whatever current you are using, and the oxidation of the copper suboxid takes place simultaneously with the deposition of the copper on this cathode. So that after you have accomplished the washing of the precipitate, which must be resorted to any case, the only thing further that remains to be done is to pour in the nitric acid and make the battery connections. At the conclusion of the test you can either draw off the liquid at the bottom by suction or else you can raise the cathode gradually out of the liquid, at the same time directing a stream of water upon it. Of course it is washed off with water and then with alcohol and at once dried and weighed. This apparatus has given very satisfactory results in our laboratory.

I did not prepare any figures in connection with this method, because in the preceding paper, to which I referred, I have given very full results that have demonstrated that by this process a very thorough solution and deposition of the copper took place, and I only made qualitative tests to assure myself of the complete deposition of the copper. I mention it at this time, thinking possibly it might be of interest to some members of the association.

There is another method on which I did a little work and had a few results, but I have not carried it sufficiently far to justify me in presenting anything definite on the subject. I might say in passing that the principle of this method was first suggested to me by a paper by Strache and Kitt in *Monatshefte für Chemie*, vol. 12, p. 380, a number of years ago. The method was applied to the determination of aldehydes and ketones. This is entirely a separate and distinct modification. I have gotten some very good results, but not enough to justify me in presenting a paper at this time. The process depends upon the fact that after we have partially precipitated the copper in Fehling's solution by means of reducing sugars, upon adding phenylhydrazine to

the solution we will reduce the remainder of the copper, and a definite amount of nitrogen will be given off, and this nitrogen can be measured. I do not know whether this same principle has already been applied, but if it has I would like to be informed on the subject.

The following equation gives an idea of the chemical changes involved:



The PRESIDENT. If there is no further discussion on the subject of sugars, the report of Committee B will be made by Mr. Winton.

REPORT OF COMMITTEE B ON RECOMMENDATIONS.

Mr. WINTON. Committee B is ready to report, first, on the methods for foods and feeding stuffs.

FOODS AND FEEDING STUFFS.

There are a great many changes suggested here. We will have to pass them very rapidly. First of all, under the head of moisture, your committee suggests that in place of the method as given we substitute the following. I do not know whether any of you have copies of Bulletin 46, or of the methods for foods and feeding stuffs as revised in 1900 and printed in Circular 7, Division of Chemistry, United States Department of Agriculture. It will be rather hard to follow these changes unless you have a copy of the methods at hand.

Under "2. Determination of moisture," for the first sentence substitute the following:

"Dry 2 grams of the substance for five hours at the temperature of boiling water in a current of dry hydrogen, in vacuo or in the air."

But as linseed meal, cotton-seed meal, and corn products contain oils subject to oxidation, we thought it would be better to insert this clause in addition: "Linseed meal, cotton-seed meal, corn products, or other substances containing oils subject to oxidation must be dried either in hydrogen or in vacuo." That is the first recommendation. The change is that the use of air be allowed, except in the case of substances containing drying oils. I move the adoption of this substitute in the method of moisture.

Adopted.

Second, under "4. Determination of Ether Extract (b) (1), Direct Method," changes are introduced to make this procedure consistent with the determination of moisture—that is to say, it is stated here to dry before determining the ether extract. We have so worded it that the method of drying will be the same used in the former case. Change the first sentence to read as follows: "Dry 2 grams of the substance as for the determination of moisture and extract with anhydrous, alcohol-free ether in a continuous-extraction apparatus for sixteen hours." There is another slight change there. We have inserted the clause, "in a continuous-extraction apparatus."

It seemed to be a little ambiguous. I move the adoption of this change.

Mr. WHEELER. I would like to ask the chairman of the committee whether Blatchford's calf food and several other mixed foods on the market contain these oils that are likely to be oxidized and how anyone is to know whether such oils are present. Suppose that we had a case of mixed food. One of my assistants inadvertently dried one in

the air, extracted the fat, and obtained 5 or 6 per cent. When it was dried in hydrogen in a proper manner we obtained nearly 12 per cent. I fear this is a very serious question and think we ought to hesitate before adopting it. I think it is unwise for us to take this action without further consideration. I move, therefore, that this be laid upon the table until the next meeting of the association.

Mr. WINTON. Do you move also that we reconsider the other vote? The two methods should be the same.

Mr. WHEELER. I think that would require the same motion to reconsider. I make a motion to lay this upon the table, and will follow with a motion to reconsider.

The PRESIDENT. The motion has been made and seconded that the recommendation as read be laid upon the table for one year.

Carried.

Mr. WHEELER. I make a motion that we rescind the vote whereby we voted to adopt the recommendation relating to moisture.

The PRESIDENT. The motion is made and seconded that we rescind the action whereby we adopted the recommendation relating to moisture.

Carried.

Mr. WHEELER. I would now move that this be laid upon the table for one year, and that this matter, as well as the matter of determining ether extract, be referred to the referee.

Mr. WINTON. I think myself that this is wise. I called the attention of the committee to the absolute necessity of drying linseed meal and some of the other products in hydrogen or else in vacuo, and it was conceded by the committee that wherever such substances were present it would be necessary to dry as we have done heretofore. The only point seemed to be that the majority of the members were using the air on ordinary substances. We ourselves use hydrogen. Our only purpose was to learn the wishes of the majority in this matter. As for Blatchford's calf food, that contains a large amount of linseed meal. Our intention was that if there was any doubt whatever, hydrogen or a vacuum would be used.

The motion was put and carried.

Mr. WINTON (continuing report of Committee B):

Under the head of diastase method for the determination of starch a few changes are recommended. First, in the *official method*^a it is recommended that we strike out the words "from" and "to 40," leaving 20cc of malt extract to be used. In order to understand this I will have to go ahead a little and point out some of the other changes. Perhaps these all might be considered together, as they touch the same matter. The kernel of the changes suggested is this: According to the official method only one digestion with malt extract is made. The material is boiled with water, then it is digested for an hour with malt extract. The German chemists and some

^aAs printed in Circular 7, Bureau of Chemistry, U. S. Dept. Agr.

of us here in America find that we can not in some materials possibly get the starch all changed into maltose by a single digestion. We first make the paste, then treat it with the malt extract. A certain amount of starch resists the rupturing in the first place and consequently resists the action of the diastase. In order to get it all in solution we heat a second time to boiling. * * * After the bulk of the starch is taken out these remaining starch granules are more readily brought into semi-solution and afterwards converted into maltose. The changes which we suggest are those which some European analysts follow and which some of us have found are absolutely necessary. Following the methods here described, I have been in some cases unable to get within 2 or 3 per cent of the amount of starch; and as this change does not affect the method except as to add an extra precaution, I recommend that it be adopted. I will read the changes: First, strike out 40cc and use 20cc the first time. Then instead of digesting with malt after thus boiling until a microscopic examination shows the residue free from starch, it is proposed that we digest for a definite length of time, and then without examining it with the microscope boil again. The microscopic examination is mentioned later in the paragraph. I think we might vote on this first. I move the adoption of these changes.

Mr. WHEELER. I rise to second that motion, but would like to ask whether the motion carries with it the retention of the microscopic examination at the end?

Mr. WINTON. I noticed that omission. As it now stands, that was taken out, but I think it was an oversight, and I am quite sure that the committee will approve of the retention of the microscopic examination later on.

Mr. WHEELER. If the committee would retain that feature, I would second the motion.

Mr. WINTON. The committee are agreed on that point; it will be put in later in the paragraph.

The motion was put and carried.

Mr. WINTON (continuing report of Committee B):

We have some suggestions as to neutralization. As stated in the methods, the acid is nearly neutralized with carbonate of soda while still hot. It is the general experience that neutralizing that solution while hot causes decomposition of some of the carbohydrates, and we therefore recommend that we cool before neutralizing, and also that instead of sodium carbonate we use a dilute solution of sodium hydrate. I think most of us do use the latter, but sodium carbonate appears to be in the method. We therefore recommend that the solution be cooled and neutralized with sodium hydrate instead of sodium carbonate, or nearly neutralized with sodium carbonate.

Mr. WHEELER. If in order, I would like to suggest that these recommendations be printed in the proceedings and that action be deferred until the next meeting of the convention. I think we came very near making a serious mistake this morning. I think there was some good reason for using the sodium carbonate. I do not feel that the members are quite in a position to act without having the methods before them. If in order, I would move that further recommendations be printed in the proceedings and that definite action be deferred until the next meeting of the association unless there is some very urgent matter.

THE PRESIDENT. Is that motion intended to apply to all of the recommendations of the committee not yet recommended?

MR. WHEELER. Unless they concern some very urgent matter.

MR. WINTON. I think this matter in connection with starch is urgent. If you do not consider that precaution you will not get your results. And as for the others, I will say that the originators of these methods and those who have done a large amount of work have used sodium hydrate. I know that Mr. Krug strongly advises it. I have always used it myself. As for getting the association into any trouble, I do not think it will. I think it is merely in the line of improving the methods. I must say, however, that notwithstanding the vote of the committee and the opinion of those whom we have consulted, notwithstanding that their opinion was that we dry in air in most cases, I must say that personally I would like to go slowly. But in these other matters it seems to me that the case is somewhat different.

MR. WHEELER. I will withdraw my motion.

MR. WINTON. I think we have here those who have had a great deal of experience in these matters; and as they are not new considerations at all—they are matters that have been thoroughly investigated—I see no reason why these changes should not be made, especially as we are about to get out a new book on the analysis of fertilizers. We think these changes ought to go into the new edition.

It was then moved, seconded, and carried that the recommendations as read by chairman of committee be adopted.

MR. WINTON (continuing report of Committee B):

The third recommendation is a very important one. I will say that not only the methods for feeding stuffs, but many of our methods, need rewriting. There are typographical errors, and there are errors that we can not lay to the printer that should be corrected, and it was the purpose of the committee to take this opportunity to change some directions here that are obviously erroneous. Now, in making up the malt extract the directions are to digest over night. Those who have tried it get a sour malt extract. The German chemists digest two or three hours, and I think those of us who have had experience do this. Some say that even half an hour is long enough, but we stick to the time-honored method. I move, therefore, that we digest two or three hours instead of overnight in preparing the malt extract.

Carried.

The next changes are under the head of the phloroglucin or glucol method. I will say that these changes have all been carefully reviewed by Mr. Krug, who, I think, has been largely responsible for these methods. What I have to present is the result of this study on his part. The first change—I do not think that this will be intelligible, because hardly any of you have this bulletin—is that the first line be made to read “A quantity of the material, chosen so that the weight of the phloroglucid obtained shall not exceed 0.300 gram, is placed in a flask.”

This change was adopted.

In the seventh line the words “by means of a separatory funnel and” are to be inserted after “added.” I will say this is all Greek to me, and it seems to be to you.

Mr. BIGELOW. As this seems to be a technical matter, which concerns a rather limited number of the workers who have all passed upon it, I suggest that the recommendations be read and adopted as a whole, unless some one wishes to raise an objection. I make a motion to that effect.

The PRESIDENT. The chairman of the committee may go ahead and read these recommendations, and then an opportunity will be given for objection or statement of opinion, and at the end a motion will be in order to adopt the whole. If there is no objection to that line of proceeding, we will do that way.

Mr. WINTON (continuing report):

In the seventeenth line the words "from three to" are to be stricken out; that is to say, the idea here is merely to confine the quantity used to the amount found best suited for the purpose.

In the eighteenth line the words "in a weighing bottle" are to be inserted after "weighed."

Under the head of "Galactan Method," in the twelfth line, the word "gently" is stricken out, and the words "at 80 degrees centigrade" inserted after "bath," and the words "with constant stirring" after "minutes." I will say that Mr. Krug explained all these matters in detail, and the changes seemed desirable ones. They are based on a great deal of experience.

In the seventeenth line the words "avoiding unnecessary heating, which causes decomposition," are inserted after "bath." The referee finds that there is danger of this if you are not cautious.

In the twenty-first line the words "three hours" are substituted for "a short time." These are all of the recommendations for changes in the methods.

Mr. BIGELOW. I move the adoption of these recommendations.

The PRESIDENT. A motion has been made and seconded that the recommendations as read be adopted. Are there any remarks?

Carried.

The committee further recommends that the referee for the ensuing year on feeding stuffs undertake a study of the König method for the determination of crude fiber. This was suggested by Mr. Fraps in his paper, and the committee recommend that the referee be requested to undertake a study of this method.

Adopted.

Mr. WINTON (continuing report):

This covers all the recommendations with regard to feeding stuffs.

SUGAR.

The committee recommend that the work on sugar be divided and that associate referees be appointed for three divisions of the work, as follows: First, optical methods; second, chemical methods, and third, special analytical methods used in the sugar industry. These are Mr. Ewell's suggestions.

Adopted.

I believe Mr. Krug has the recommendations in regard to tannin.

Mr. KRUG. I do not believe that it is necessary to read these all in detail. There are a number of minor changes in the methods, merely in the wording, to cover the recommendations of the referee. The dilution is changed from 0.8 gram per 100 cc to a constant quantity of tannin, the amount of material to be taken being varied. These are minor changes and are submitted for incorporation in the printed report.^a

In "soluble solids," paragraph 5, the whole paragraph has been completely rewritten, as follows:

Double pleated filter paper (S. and S., No. 590, 15 cm) shall be used. To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, and decant as much as possible. Add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc of filtrate, evaporate the next 100 cc and dry. Evaporation during filtration must be guarded against.

The optional method, which was a double precipitation of lead acetate, is stricken out.

In paragraph 6, on nontannins, the line beginning with the word "prepare" and ending with the word "linen" is stricken out and the following is substituted:

Prepare 20 grams of hide powder by digesting twenty-four hours with 500 cc of water and adding 0.6 gram chrome alum in solution, this solution to be added as follows: One-half at the beginning and the other half at least six hours before the end of the digestion. Wash by squeezing through linen, continue the washing until the wash water does not give a precipitate with barium chlorid.

Throughout the method, in agreement with the recommendations of the referee, 100 cc has been substituted for 50 cc to allow for evaporation.

In the determination of nontannins the following provisional method is recommended to be added:

To 14 grams of dry chromed hide powder in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently; shake fifteen minutes; throw on funnel with a cotton plug in the stem, let drain, tamp down the hide powder in the funnel, return the filtrate until clear, and evaporate 100 cc.

These cover all the changes in the methods.

The committee further recommends that the referee for the ensuing year be instructed to investigate methods for the determination of the acidity of tan liquors. This last recommendation is in the line of expansion. The gentlemen who are especially interested in these analytical methods feel that the determination of the tannin is in very good shape at present; the method is quite exact; the work shows good results, and they would like to have the association take up a new line. The methods that are at present used in the various laboratories in the determination of such a simple matter as the total organic acids, which are formed by fermentation in the liquors used in the tannery, are of such a varied character that we think the time has come when some method should be tried and worked out, and the various methods that have been used should be compared and the best one chosen for this work.

Adopted.

^a These minor changes were as follows:

II. Quantity of material: First line, substitute 0.35 to 0.45 for 0.8.

Second line: Substitute "tannin" for "solid."

Sixth line: Substitute 0.35 to 0.45 for 0.8, and "tannin" for "solids."

III. Moisture. (b) 1: Substitute "eight" for "twenty-four."

(b) 2: Substitute "six" for "eight;" "100° C." for "100° to 110° C."

IV. Total solids: For 50 cc read 100 cc.

Mr. WINTON. I understand there are no recommendations with regard to the methods for dairy products. Am I right?

The PRESIDENT: The only recommendations are for the continuation of work along similar lines.

Next in order is the report on food adulteration, by Committee C, which will be presented by Mr. Bigelow:

REPORT OF COMMITTEE C ON RECOMMENDATIONS.

FOOD ADULTERATION.

There are three recommendations. First, regarding the designation of the referee. In former years the subject was called fermented and distilled liquors, although the association never took any interest in distilled liquors. Some methods were offered in 1896, adopted provisionally, and never taken up again. Fermented liquors are never studied, with the exception of wines. Now that the subject of liquors is only one division, and perhaps a relatively unimportant division of the whole subject which the referee covers, the committee recommends that the title be changed to "referee on food adulteration." I would move that this recommendation be adopted.

Adopted.

Regarding the reports made by the referee, it was stated that they were considered by all of the referees together, and also in the discussion a number of other food chemists took part. The discussion occupied two days and was quite complete. The committee recommends that the methods approved by the referee and associates, which are the circulars which have been distributed, with certain corrections that have been made owing to criticisms, be printed in a separate bulletin as provisional methods.

Adopted

The PRESIDENT. Has the abstract committee any report to make?

Mr. ALLEN. I have a very short report.

REPORT OF COMMITTEE ON ABSTRACTS.

MR. PRESIDENT AND MEMBERS OF THE ASSOCIATION: The committee on abstracts appointed by the incoming president included the members of the committee of the previous year so far as they desired to continue the work. Correspondence on the part of the chairman developed the fact that several members of last year's committee were not in position to keep up the work, and one other member had gone out of official work. Some difficulty was had in securing suitable recruits to the committee, and at the suggestion of the president of the association the number of members was reduced to five instead of nine, as formerly. This reduction in number, while it imposed a larger amount of work on the individual members of the committee, simplified the laying out of the work for the year and its management, as formerly no little trouble had been experienced in directing the work of the large and widely-scattered committee and keeping it up to date.

As in previous years, the work was divided by journals instead of by subjects, the former seeming to be the only practicable method, although it is recognized that an assignment by subjects might add to the interest of the individual members of the committee. The committee has attempted to follow up regularly twenty-seven of the most prominent journals containing articles on agricultural chemical methods,

and has included, besides, abstracts from a long list of other journals which contain occasional articles in that field. The results of this work have been published as formerly in the Experiment Station Record from month to month during the year. Despite the reduction in the size of the committee, it is hoped that the scope and efficiency of the review has not suffered seriously.

E. W. ALLEN,
W. H. BEAL,
C. B. WILLIAMS,
W. F. HAND,
D. W. MAY,
Committee.

MR. KILGORE. Mr. Chairman, it was suggested by you in your address, I suppose upon considerations which came from the chairman of this committee, that the committee be discontinued. I would like to know if Mr. Allen is really of the opinion that the work could be done more efficiently by the regular editorial staff of the Record. If so, I think it would be well to adopt this report and discharge the committee. I do not think our men are looking for work that is really not necessary for them to do.

MR. ALLEN. The conditions have changed somewhat from those that existed at the time the abstract committee was first suggested. For a number of years that committee aided quite materially in following up the literature relative to methods of agricultural analysis; but in the meantime the force in our office has grown considerably and has included among its additions a number of men who were familiar with the literature of agricultural chemistry and have followed up that literature. Now, in principle such a committee would seem to be of a great deal of assistance, and in the past that has been true. But the difficulty has been each year to get men who are willing to give their time to follow this literature regularly, and unless the work is done regularly a man soon falls behind and it becomes difficult to handle it, so that abstracts which treat the literature uniformly are rather difficult to obtain. I can understand this. I do not make it as any criticism on the members who have been appointed on the committee. Seasons come in the year when the work presses unusually hard in the laboratory, and if the work is allowed to lag it becomes more difficult to take it up. It has happened that after good men have been in this work for a year or two, until they have become thoroughly familiar with it, they have lost interest and wanted to be replaced by other members. The result has been that there has been a diminution in the number of the members and an attempt to get members who are nearer at hand here in Washington. The last year there were only two members of the committee we were able to obtain who were located outside of the Office of Experiment Stations. I feel now that we are in a position in the Office to follow up that literature with reasonable regularity and completeness. We are anxious, of course, in our own publications,

to review all important articles relating to methods of agricultural analysis, and it becomes necessary, in order that we shall not overlook any important article, to open a ledger account with each man on the journals that are assigned to him, and that involves a good deal of work. Some man in the office must go over each number of this journal as they come in and make a note of those articles, which is kept as a matter of record; and if those articles are not abstracted in the course of a couple of months, he must undertake to abstract them. In that way we have sometimes delayed the abstracting, and it has made a great deal of work. So that while I appreciate very highly the work which the members of the committee have done, I believe now that the time has come when we can handle that work to the satisfaction of the association in the office.

Mr. KILGORE. We have approved the idea of expansion here this morning. To be on the safe side, I move that the association now approve the method of concentration, and that we accept this report and discharge the committee and concentrate the work in the Office of Experiment Stations.

The PRESIDENT. The Chair would suggest that you use the word "discontinue."

Mr. KILGORE. I accept the change.

The PRESIDENT. A motion has been made that the report be approved and the committee discontinued.

Carried.

The PRESIDENT. Has the committee on volumetric standards any report to make?

Mr. KILGORE. The committee has not had a meeting. The object for which this committee was appointed has been accomplished. It was to try to bring about a uniform standard with reference to the unit of volume as well as the graduation. As we all know, at the last Congress a bill was passed establishing a national standardizing bureau, and I think this association had great weight in bringing this about. I feel, therefore, that the work for which this committee was appointed has been done, and I should think that the committee might be discontinued.

Mr. EWELL. I would like to emphasize the fact that the work of this association and its members, particularly the chairman of the committee, who has just spoken, was very helpful indeed in bringing about this result, and I feel that the result and what the committee accomplished is an assurance that it is a good thing for scientists to tell legislative bodies what they want. It is not lobbying, in the accepted sense of the word. This is an excellent demonstration that Congress is ready to do what is wanted when the need is put before them in that light. This is the plan on which all the work of the standards bureau was done.

The PRESIDENT. Shall any action be taken on the suggestion of the chairman of the committee on volumetric standards?

Mr. WHEELER. I move its adoption and that the committee be discontinued.

Adopted.

The PRESIDENT. Mr. Frear, the chairman of the committee on food standards, has requested that this statement be read as a report of progress:

REPORT OF COMMITTEE ON FOOD STANDARDS.

Reports on the following subjects are ready for submission, presenting tentative food definitions and standards for criticism and suggestion before final adoption:

Meat products, lard, milk and milk products, flavoring extracts, spices, fruit preserves, etc., sugars and sirups, fruit juices, fermented liquors, tea, coffee, and cocoa preservatives.

Dr. J. B. Weems, of Iowa, has performed for the committee a considerable amount of research work in the study of the constants of the raw materials used in the manufacture of butter substitutes in America.

WM. FREAR, *Chairman*.

The report was adopted.

The PRESIDENT. We will now hear from the committee on resolutions. Mr. Wheeler has the floor.

REPORT OF COMMITTEE ON RESOLUTIONS.

The following resolution was presented by Mr. Ewell:

Resolved, That the title "Determination of nitrogen" be changed to read "Determination of nitrogen and nitrogenous bodies," and that the work on nitrogen be divided between two or more referees. Further, that the nitrogen methods be taken out of fertilizer methods and proper reference inserted in their place.

The committee recommend that this resolution be referred to the referee on nitrogen, with suggestions as to titles, etc., a definite report to be made at the next meeting of the association.

Mr. KILGORE. I move the adoption of the report of the committee, which will carry a reference of this to the nitrogen referee for a report at the next meeting.

Seconded.

Mr. EWELL. I move to amend by adding that the executive committee be instructed to consider the advisability of appointing two referees, one for methods for the determination of nitrogen, and the other for the separation of nitrogenous bodies for their determination, so that we may start the work between now and the next meeting.

Seconded.

The PRESIDENT. The amendment is that the executive committee be instructed to consider the subject of the appointment of referees in connection with the division of nitrogen work—that is, I suppose, a division on the lines laid down in the original motion.

Mr. WHEELER. I think that is perfectly satisfactory. The reason for laying over the general matter was that there might be an orderly and systematic consideration of the whole matter.

Amendment carried. Original motion as amended carried.

Mr. WHEELER. The next is a resolution introduced by Mr. Ross:

Resolved, That the president-elect appoint, during the future sittings of this convention, the committees on recommendations of referees who are to serve during the next succeeding annual convention.

The idea of this is that the men who are to serve on these committees will have notification and will be able to look up the matters upon which they are to pass judgment and come here better prepared to fill the positions than were they notified only after their arrival. I move the adoption of the resolution.

Carried.

The PRESIDENT. Has the committee on resolutions anything further?

Mr. WHEELER. That is all concerning resolutions. If it is in order, I would like to introduce a motion affecting the future policy of the association to a certain extent. I move that the committees on recommendations of referees shall meet during or at the close of the next annual convention and prepare their reports upon the recommendations of the referees, the same to be distributed to the members of the association at least six months before the next succeeding annual convention; provided, however, that by a two-thirds vote of the voting members present at any session the above-mentioned committee may be instructed to report during the same annual convention upon minor matters and matters of urgent necessity, and that action of the association may be taken thereon without delaying until the subsequent annual convention.

This provides that the association may, at the next annual convention, or at any annual convention, act upon matters of minor importance or of urgent necessity immediately; but in view of the fact that we assume to act upon a large number of new methods, particularly in connection with the food investigation, it was thought wise to make some provision by which all the proposed methods and all the important changes should be placed in the hands of every member of the association, so that he can look up the question and come here prepared to vote intelligently. I move that this be adopted.

Seconded.

Mr. KILGORE. Our constitution already provides that no matter shall be passed upon by the association with reference to the change in a method until it has been submitted to the test of the members of the association. It appears to me that this resolution concerns a matter which is fixed by the constitution, and I think that either the constitution ought to be changed so as to take in this matter, or else the resolution ought not to be passed.

The PRESIDENT. The constitution refers to those materials that are affected by inspection. It would be those materials that, as things stand now, would not be affected by this resolution.

Mr. WHEELER. This motion, as I understand it, is not in collision at all with that provision in accordance with which the methods are to be tested before adoption, but merely provides that all of the recommendations of the committee on recommendations of referees shall be distributed to each member of the association, so that, before the final vote is taken upon the adoption of these methods and upon important modifications, each member shall have seen the final report of these committees on the recommendations. We have seen to-day the fact that the body of the house is not in position to know just what these changes mean, because we have not the methods before us. I consider this is in nowise in conflict with the constitution. It only provides that every member shall be furnished with important recommendations.

Mr. KILGORE. I do not object to the resolution at all. The spirit and intent of it are good. But it would make a delay of two years before any change in a method could be brought about, instead of one year as provided now, and the constitution gives us the right to make a change within one year provided the test has been satisfactory and two-thirds of the association so vote.

Mr. WHEELER. I fear the motion is not understood. It was intended to provide that matters which arise next year, which under our constitution can not be adopted until the year following, may be adopted the year following just the same; but that before we come here to vote upon them we shall have a printed list of those recommendations. This does not delay the adoption, and provides that by a two-thirds vote we may act upon anything which is of immediate necessity—minor matters or matters of urgent necessity—and that all others will be acted upon just as early as they would be under our constitution.

Mr. HOPKINS. It seems to me that the machinery to accomplish these results is rather cumbersome. Would it not be possible to have the referee by circular letter notify the members of the association a month or so in advance of the meeting of the proposed changes, thus referring it, as you say, to a committee of the whole?

Mr. WHEELER. That plan has been considered by a number of the members of the association, and the trouble with that is that the recommendation of the referee is not the final recommendation upon which this association has to act. We are obliged to act upon the recommendation of the committee, and the referee is not in position to render any such report beforehand.

The PRESIDENT. I believe a motion has been made and seconded that the resolution as read be adopted.

Carried.

The PRESIDENT. Has Mr. Wiley any report to make for the committee on fertilizer legislation?

Mr. WILEY. The committee has not taken any action in regard to any fertilizer legislation, and it does not seem to me necessary to continue this committee any longer. If anyone thinks the committee should be continued of course it can be done, but it is hardly necessary to carry a committee unless there is something for it to do. The fact is that the fertilizer laws in the States are so different that it is almost impossible to draw any general scheme which will meet the approbation of any considerable number of States. If there could be a national fertilizer law regulating interstate commerce in fertilizers then the committee would have a *raison d'être*, because it could do some good work. Such a law would be desirable. But those of you who know the difficulty of even getting a national pure-food law may realize how remote the prospect is of getting one controlling interstate traffic in fertilizer materials.

It may be interesting for you to know that the President of the United States is interested in pure-food legislation to such an extent that he has asked the Secretary of Agriculture for an expression of opinion as to what steps the Executive should take in the matter, and it is more than likely that we shall have an expression from the President as to the advisability of Congress taking some action along this line.

I would like to hear from some of the gentlemen present who are directly in contact with State legislation as to the desirability of continuing this committee.

Mr. KILGORE. I feel that the committee should be continued. In the South we have been able to accomplish something. Our commissioners of agriculture of the cotton States have an organization, and through that organization we have drafted what we call a uniform fertilizer law for the States connected with the association. The legislature of the State of Georgia is now wrestling with that bill. The fact that this association has a committee on the subject has been helpful with us in getting the matter to the attention of the proper parties concerned, and for the moral effect of it at any rate we would like, in my section of the country, to see this committee continued.

Mr. WHEELER. I will say that a similar committee appointed by the Association of Agricultural Colleges and Experiment Stations has this year brought the matter before the United States commission on uniform legislation, and at their last meeting at Denver they instructed their members in the respective States to use their efforts, in cooperation with the committee of this association and the colleges and experiment stations, to secure modifications of laws in their respective States in accordance with this general scheme; and I feel that merely for the sake of keeping before our members the fact that there is a scheme after

which it is hoped the laws of the individual States will be modeled, this committee should be continued.

Mr. KILGORE. I move that the report be approved and the committee continued.

Carried.

The PRESIDENT. I would say, in closing, that this has been in attendance the largest convention that the association has ever held. Last year the attendance was 85, which was the largest up to that time. This year the attendance is well on toward 100, and I think that the interested manifested in all of the sessions of the meeting, both by the number present and by the close attention given to the proceedings, indicate a state of thorough interest and support in the work of this association. In some respects this meeting has been an exceptional one aside from the large attendance. We have all noticed the comparatively small amount of time called for by discussions. This has been owing to the nature of the work which happened to be of a peculiar character this year. A large amount of new work came before the association which was in a measure pioneer in its character, and work which had to be largely considered and digested in committee outside of the convention's sessions. So that there has been a very much smaller amount of discussion than would have been called forth had we proceeded in the manner which we employed some years ago in bringing everything, even in embryo, onto the floor of the convention. For illustration, the committee on food adulteration, of which Mr. Bigelow was chairman, has been at work very busily this last year, and by the employment of the most admirable method which Mr. Bigelow used in getting the reports of the members of the committee printed and sent out for inspection and criticism, and then by having the associate referees of that committee hold meetings for the best part of two days here in Washington before and during this convention, this enormous mass of matter has been put before the convention in such a shape as to require a very small amount of time. Of course the time for discussion will properly come when these methods have been still further worked upon and digested and submitted to the future conventions of this association for final action. It seems to me that the association has made very great progress in the matter of economizing time. To those who have attended the convention of this association for the first time this year I would say, therefore, that this meeting has not been a typical one in every respect, but rather exceptional in point of the small amount of discussion, owing to the peculiar conditions that have prevailed, and in general to the comparative shortness of the session. It is not to be assumed, I trust, that our conventions hereafter will be necessarily as short. I am sure we can not be charged with any undue haste, nor can we be charged with undue delay in the proceedings.

I took pains in my opening address not to express any special gratitude to the association for having placed me in the position of presiding officer, for the reason that I felt that I could a good deal better and more truthfully express my feelings at the close of the convention than I might at the beginning. I would say now that I am grateful to the association for the ease with which they have made it possible for the chairman to preside at the sessions of the convention. The attention has been uniformly close and the behavior of the members has been remarkable.

Mr. KILGORE. I would like to offer this verbal resolution:

Resolved, That the authorities of the Columbian University have placed our association under renewed obligations to it by furnishing us facilities for our meeting, and that the sincere thanks of the association are extended to the authorities for this continuance of their favor.

I move the adoption of the resolution.

Carried.

Mr. WHEELER. I move that a hearty vote of thanks on behalf of the association be extended to the Cosmos Club for their courtesy, and a similar vote of thanks to the Secretary of Agriculture for his patronage and assistance by which we have been able to have the reports here for distribution; and that the secretary be instructed to transmit to the secretary of the Cosmos Club and to the Secretary of Agriculture the expression of the thanks of this association.

Mr. KILGORE. And I would like to have included in the first resolution that the secretary furnish a copy to the authorities of Columbian University.

Mr. WHEELER. I am glad to include that in my motion.

Carried.

Mr. ROSS. I move that the thanks of this association be tendered to the retiring president for the able, impartial, and courteous manner in which he has presided over its deliberations.

Carried.

At 11.15 a. m. the convention adjourned sine die.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1902.

PRESIDENT.

Mr. H. J. WHEELER, Kingston, R. I.

VICE-PRESIDENT.

Mr. R. J. DAVIDSON, Blacksburg, Va.

SECRETARY.

Mr. H. W. WILEY, Washington, D. C.

ADDITIONAL MEMBERS OF EXECUTIVE COMMITTEE.

Mr. C. G. HOPKINS, Champaign, Ill.

Mr. F. D. FULLER, Geneva, N. Y.

REFEREES.

Phosphoric acid: C. H. Jones, Burlington, Vt.

Nitrogen:

Determination of nitrogen: Fred W. Morse, Durham, N. H.

Separation of nitrogenous bodies: L. L. Van Slyke, Geneva, N. Y.

Potash: H. B. McDonnell, College Park, Md.

Soils: F. P. Veitch, Washington, D. C.

Dairy products: George Cavanaugh, Ithaca, N. Y.

Foods and feeding stuffs: C. A. Brown, jr., State College, Pa.

Food adulteration: W. D. Bigelow, Washington, D. C.

Sugar: G. L. Spencer, Washington, D. C.

Tannin: Wm. H. Teas, Ridgway, Pa.

Insecticides: J. K. Haywood, Washington, D. C.

Ash: G. S. Fraps, Raleigh, N. C.

ASSOCIATE REFEREES.

Phosphoric acid: B. H. Hite, Morgantown, W. Va.

Nitrogen:

Determination of nitrogen: Edward B. Holland, Amherst, Mass.

Separation of nitrogenous bodies: R. Harcourt, Guelph, Canada.

Potash: Henri D. Haskins, Amherst, Mass.

Soils: C. C. Moore, Washington, D. C.

Dairy products: C. A. Brown, jr., State College, Pa.

Foods and feeding stuffs: F. D. Fuller, Geneva, N. Y.

Food adulteration:

- (1) Meat and fish: W. D. Bigelow, Washington, D. C.
- (2) Fats and oils, not including dairy products or their substitutes: L. M. Tolman, Washington, D. C.
- (3) Cereal products: A. McGill, Ottawa, Canada.
- (4) Infants' and invalids' foods: H. W. Wiley, Washington, D. C.
- (5) Saccharine products, including confectionery: A. E. Leach, Boston, Mass.
- (6) Vegetables—canned, dried, or otherwise preserved: L. S. Munson, Washington.
- (7) Tea and coffee: W. H. Ellis, Toronto, Canada.
- (8) Cocoa: F. T. Harrison, London, Ontario.
- (9) Spices and condiments: A. L. Winton, New Haven, Conn.
- (10) Vinegar: Wm. Frear, State College, Pa.
- (11) Flavoring extracts: A. S. Mitchell, Milwaukee, Wis.
- (12) Fruit products: L. M. Tolman and L. S. Munson, Washington, D. C.
- (13) Fermented and distilled liquors: W. D. Bigelow, Washington, D. C.
- (14) Baking powders and baking powder chemicals: A. L. Winton, New Haven, Conn.
- (15) Preservatives: W. M. Allen, Raleigh, N. C.
- (16) Dyes: L. M. Tolman, Washington, D. C.

Sugar:

Optical methods: L. M. Tolman, Washington, D. C.

Chemical methods: L. S. Munson, Washington, D. C.

Special analytical methods used in sugar industry: D. S. Davol, Caro, Mich.

Tannin: Geo. A. Kerr, Damascus, Va.

Insecticides: James Emory, Washington, D. C.

Ash: F. T. Shutt, Ottawa, Canada.

COMMITTEE ON FOOD STANDARDS.

Mr. Wm. Frear, State College, Pa., Chairman.

Mr. H. W. Wiley, Washington, D. C.

Mr. H. A. Weber, Columbus, Ohio.

Mr. M. A. Scovell, Lexington, Ky.

Mr. E. H. Jenkins, New Haven, Conn.

COMMITTEE ON FERTILIZER LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., Chairman.

Mr. B. W. Kilgore, Raleigh, N. C.

Mr. H. B. McDonnell, College Park, Md.

Mr. H. A. Huston, Lafayette, Ind.

Mr. B. B. Ross, Auburn, Ala.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members *ex officio* and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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